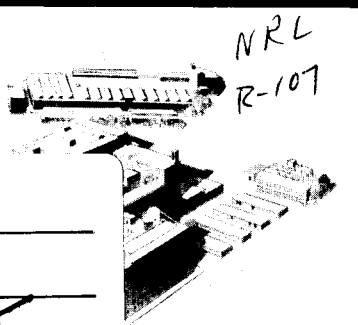


GPO PRICE \$ \_\_\_\_\_  
 CFSTI PRICE(S) \$ \_\_\_\_\_  
 Hard copy (HC) 3.00  
 Microfiche (MF) .50

ff 653 July 65



FACILITY FORM 802

N 66-12968  
 (ACCESSION NUMBER)  
56  
 (PAGES)  
 CR-68310  
 (NASA CR OR TMX OR AD NUMBER)

(THRU)  
1  
 (CODE)  
23  
 (CATEGORY)

BAUSCH & LOMB INCORPORATED  
 ROCHESTER 2, NEW YORK

Grating Groove Formation in Au and Au-Ge Alloys  
 Final Report on Task 1 of Extension  
 NONR-4277 (00) (X) Amend #1

Submitted by  
 Bausch & Lomb Incorporated  
 September 25, 1965

C. Frank Mooney  
 Grating Research Section

## DOCUMENT CONTROL DATA - R&amp;D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

|  |                              |   |  |
|--|------------------------------|---|--|
| 1. ORIGINATING ACTIVITY (Corporate author)<br>Grating Research Section<br>Bausch & Lomb Incorporated<br>Rochester, New York 14602  |                              | 2a. REPORT SECURITY CLASSIFICATION<br>Unclassified  |  |
| 3. REPORT TITLE<br>Grating Groove Formation in Au and Au-Ge Alloys,<br>EXT.1 Task 1 - Surface Polish Effects on Au Mirrors   |                              | 2b. GROUP _____   |  |
| 4. DESCRIPTIVE NOTES (Type of report and inclusive dates)<br>EXT. 1, Task 1 - Final - April 1, 1965-September 30, 1965   |                              |   |  |
| 5. AUTHOR(S) (Last name, first name, initial)<br>Mooney, C. Frank  |                              |   |  |
| 6. REPORT DATE<br>September 30, 1965   | 7a. TOTAL NO. OF PAGES<br>56 | 7b. NO. OF REFS<br>7  |  |
| 8a. CONTRACT OR GRANT NO. <i>NASA order R 107/57/25</i><br>NONR 4277(00)(X) Amend #1<br>b. PROJECT NO.<br>ONR:630:JR:gc<br>c. Req.No. 00173-4-006077 Rev #2<br>d. 12-31-64   |                              | 8b. ORIGINATOR'S REPORT NUMBER(S)<br>DD 1473 - B/L-1<br>8c. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) |  |
| 10. AVAILABILITY/LIMITATION NOTICES<br>Qualified requesters may obtain copies of this report from <i>NASA</i> <del>DD</del> .  |                              |   |  |
| 11. SUPPLEMENTARY NOTES<br>Scientific Officer: Dr. Richard<br>Washington, D.C. Tousey, NRL<br>20390  |                              | 12. SPONSORING/MONITORING ACTIVITY<br><i>NASA</i><br>Office of Naval Research<br>Washington, D.C. 20360 <i>12968</i>                |  |
| 13. ABSTRACT This report has two principal parts. Part I reports a study of the influence of technique procedures on the appearance of electron micrographs of mirror surfaces. Part II reports a study both by electron microscopy and by vacuum ultraviolet reflectometry of effects ascribable to surface polish. The Introduction describes the equipment and procedures that were used in a standardized fashion throughout the work reported.<br><br>The important result of Part I is the evidence that any object that can be seen to have a shape in one of our electron micrographs is not some artifact of the electrons, the C-film substrate, the Pt-C shadow deposit, or the photographic practice. A change of earlier procedure was to avoid using tape in lifting replica specimens.<br><br>The most important result of Part II is that transfer films of gold have many times higher reflectance than thick (.4 micron) first surface gold films in the XUV region of the spectrum. There is further evidence, apparently significant, that transfer mirrors made from glass polished under liquid are better than conventionally polished ones.<br><br><i>Author</i> |                              |   |  |



# Security Classification

| 14. KEY WORDS       | LINK A |    | LINK B |    | LINK C |    |
|---------------------|--------|----|--------|----|--------|----|
|                     | ROLE   | WT | ROLE   | WT | ROLE   | WT |
| Diffraction Grating |        |    |        |    |        |    |
| Electron Microscope |        |    |        |    |        |    |
| Gold                |        |    |        |    |        |    |
| Mirror              |        |    |        |    |        |    |
| Polish              |        |    |        |    |        |    |
| Vacuum Ultraviolet  |        |    |        |    |        |    |
| XUV                 |        |    |        |    |        |    |

## INSTRUCTIONS

1. **ORIGINATING ACTIVITY:** Enter the name and address of the contractor, subcontractor, grantee, Department of Defense activity or other organization (*corporate author*) issuing the report.

2a. **REPORT SECURITY CLASSIFICATION:** Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.

2b. **GROUP:** Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.

3. **REPORT TITLE:** Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parenthesis immediately following the title.

4. **DESCRIPTIVE NOTES:** If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.

5. **AUTHOR(S):** Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.

6. **REPORT DATE:** Enter the date of the report as day, month, year; or month, year. If more than one date appears on the report, use date of publication.

7a. **TOTAL NUMBER OF PAGES:** The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.

7b. **NUMBER OF REFERENCES:** Enter the total number of references cited in the report.

8a. **CONTRACT OR GRANT NUMBER:** If appropriate, enter the applicable number of the contract or grant under which the report was written.

8b, 8c, & 8d. **PROJECT NUMBER:** Enter the appropriate military department identification, such as project number, subproject number, system numbers, task number, etc.

9a. **ORIGINATOR'S REPORT NUMBER(S):** Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.

9b. **OTHER REPORT NUMBER(S):** If the report has been assigned any other report numbers (*either by the originator or by the sponsor*), also enter this number(s).

10. **AVAILABILITY/LIMITATION NOTICES:** Enter any limitations on further dissemination of the report, other than those

imposed by security classification, using standard statements such as:

- (1) "Qualified requesters may obtain copies of this report from DDC."
- (2) "Foreign announcement and dissemination of this report by DDC is not authorized."
- (3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through \_\_\_\_\_."
- (4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through \_\_\_\_\_."
- (5) "All distribution of this report is controlled. Qualified DDC users shall request through \_\_\_\_\_."

If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.

11. **SUPPLEMENTARY NOTES:** Use for additional explanatory notes.

12. **SPONSORING MILITARY ACTIVITY:** Enter the name of the departmental project office or laboratory sponsoring (*paying for*) the research and development. Include address.

13. **ABSTRACT:** Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as (TS), (S), (C), or (U).

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. **KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.

Grating Groove Formation in Au and Au-Ge Alloys

Final Report on Task I of Extension I

Surface Polish Effects - Au Mirrors

Sponsored by: The Office of Naval Research

(Naval Research Laboratory)

NONR-4277 (00)(X) Amend #1 *under NASA Order R-407/09 1/9/65*

ONR:630:JR:gc

Req. No. 00173-4-006077 Rev #2

12-31-64

Research by: Grating Research Section

Bausch & Lomb Incorporated

April 1, 1965 to September 30, 1965

C. Frank Mooney

Reproduction in whole or in part is permitted for any purpose  
of the United States Government.

## CONTENTS

|   |    |
|---|----|
| TASK I - DEFINITION FROM CONTRACT PROPOSAL  | 1  |
| ABSTRACT  | 2  |
| INTRODUCTION  | 3  |
| <br>  |    |
| PART I - ELECTRON MICROSCOPY OF MIRROR SURFACES   | 9  |
| <br>  |    |
| 1. Specimen Film Material Experiments   | 9  |
| 2. Shadow Obliquity Angle Experiments   | 13 |
| 3. Shadow Film Material Experiments   | 17 |
| <br>  |    |
| PART II - SURFACE POLISH EFFECTS  | 21 |
| <br>  |    |
| 1. Electron-Micrographs of Uncoated Surfaces  | 22 |
| 2. Electron-Micrographs and XUV Reflectance of First<br>Surface .4 $\mu$ Au Films                             | 30 |
| Table I. First Surface Reflectances   | 31 |
| 3. Electron-Micrographs and XUV Reflectance of Au (Cr) Films<br>Transferred from Surfaces of Differing Polish | 36 |
| Table II. Transfer Mirror Reflectances  |    |
| 4. Reflectometer Accuracy   | 43 |
| <br>  |    |
| PART III - FLASH COATED TRANSFER MIRRORS  | 45 |
| <br>  |    |
| PROGRESS ON TASK 2 OF CONTRACT EXTENSION  | 51 |
| Table III. Table of Transfer Cements  | 52 |
| <br>  |    |
| REFERENCES  | 53 |

## TASK I - DEFINITION FROM CONTRACT PROPOSAL

### "Surface Texture Effects"

"The test methods that will be used are spectral reflectivity and electron microscopy measurements. Artifacts of electron microscopy procedures must be studied. Surfaces that will be analyzed include: A liquid, Mica, Fire-polished glass, and both gold and platinum flash coated mirrors.

"Transfer coat mirrors made with familiar cement systems will be examined for efficiency, as a function of deposition conditions and as a function of master surface texture.

"Work on this task will be started as soon as contract extension arrangements have been completed, and it will continue for six months."

## ABSTRACT

This report has two principal parts: Part I reports a study of the influence of technique procedures on the appearance of electron micrographs of mirror surfaces. Part II reports a study both by electron microscopy and by vacuum ultraviolet reflectometry of effects ascribable to surface polish. The Introduction describes the equipment and procedures that were used in a standardized fashion throughout the work reported.

The important result of Part I is the evidence that any object that can be seen to have a shape in one of our electron micrographs is not some artifact of the electrons, the C-film substrate, the Pt-C shadow deposit, or the photographic practice. A change of earlier procedure was to avoid using tape in lifting replica specimens.

The most important result of Part II is that transfer films of gold have many times higher reflectance than thick (.4 micron) first surface gold films in the XUV region of the spectrum. There is further evidence, apparently significant, that transfer mirrors made from glass polished under liquid are better than conventionally polished ones.

## INTRODUCTION - ELECTRON MICROSCOPY OF OPTICAL SURFACES

### Electron Microscope

The electron microscope used for grating and film studies is a Phillips Electronic Instruments 75C. A brochure describing its features carries the number RC468 5M-U865. This equipment is operated at full voltage (75kV), maximum current, and highest magnification (3500X at the film plane).

According to "Tech Bits", 65-2, Kodak Periodical P-3,

"... most photographic materials do record almost every incident electron . . . ." Also, "If the statistical fluctuations in the electron beam could be eliminated, the ultimate limit of information storage in any material would be set by its spread function. An electron will be scattered laterally in the emulsion (along a random, irregular path), and the spread function is a measure of the mean projected area over which its energy is expanded. Two narrow electron beams that are separated by a distance much less than the radius of this area will not be resolved separately in the record.

"The spread function is not dependent upon the grain size of the emulsion (as it is for light exposures), but it does depend upon the gelatine content of the emulsion and on the emulsion thickness. The higher the electron energy, the larger the spread function. For the materials covered in this article, the spread function is of the order of 5 to 10 $\mu$  for 50 to 100kV electron exposure."

The resolving power of the 75C Electron Microscope is "30A and better" according to the manufacturer, and this has been confirmed. This figure times 3500X gives a product near  $10\mu$ . A photographic spread function of  $10\mu$  therefore does not seriously impair the resolution of the microscope.

### Photography

The photography for the electron-micrographs has been done entirely in the Grating Research Laboratory. The original pictures have been taken on Fine Grain Positive 35mm film and developed with Dectol D-72, 1:2. The film exposure to the electron beam is adjusted empirically, and several exposures are customarily taken for later selection of the best contrast. The prints are enlarged to 30,000X on Polycontrast F-paper. They are reproduced for the report by multilith.

The enlargement of the negatives to 30,000X is well within the 20X enlargement reported by "Tech Bits" 65-2 as the largest useful.

"Tech Bits" 65-2 reports:

"On subject of limitations in electron imagery, we say that all photographic materials are about equal in information-detecting ability, and no really significant improvements appear possible."

In essence, the signal-to-noise is limited by the inherent randomness in discrete event or quantum processes. If the quantum efficiency of one film is slightly inferior to another, a longer exposure restores the information content but at some risk ". . . of blurring from image motion."

Blurring due to image motion was not previously a problem to us because from the time of initial installation, the microscope has rested on a plywood sheet that is supported by four inner tubes (for trailer tires) inflated to about 3 lbs. (our construction and design.) But early in this work, the air loss allowed contact of the support sheet with the support frame. Supplementary inflation restored the insensitivity to high frequency vibration that affects the electron lens positions.

#### Specimen Support Film

The specimens used in the electron microscope consist of a carbon film that is supported by a 300 mesh Cu screen that is a standard item for electron microscopy. An electron-micrograph made through an open hole in a 300 mesh screen with the electron microscopy and photography described above is given in Fig. 1.

An electron-micrograph made through an unshadowed C film supported by a 300 mesh Cu screen, using the electron microscopy and photography described above is given in Fig. 2. The C film was prepared by



normal deposition in vacuum onto a collodion film surface that was made by floating a 1% solution of collodion in amyl acetate on water. The collodion was removed after the C film deposition by dissolving it in amyl acetate.

These basic procedures are described, for example, in The Encyclopedia of Microscopy by Clark.

#### Primary Replica Film Used to Receive Shadow

The substrate film for all shadow depositions in this report (except one on PVA and two on collodion) was a transfer film of (Mg + Al) solidly cemented to glass, skived away from the glass, and dissolved after shadowing and carbon film deposition. A reference to the details of this procedure is Anderson, Griffin, Mooney, and Wiley (Applied Optics, 4, 999, August 1965). A major modification of that procedure has been to avoid using tape for lifting replicas and specimens.



1 $\mu$

Fig. 1. Electron-micrograph made through an open hole in a 300 mesh Cu screen. The screen the electron microscope settings, and the photographic practices were nearly the same as those for all of the other electron-micrographs made for this study.

Photographic Negative 199-23.



1 $\mu$

Fig. 2. Electron-micrograph made through an unshadowed carbon film supported on a Cu screen. The film was made by vacuum deposition on collodion which in turn was made by floating a 1% solution in amylacetate on water. The collodion was dissolved away before the electron microscopy.

Photographic Negative 218-34.

## PART I ELECTRON MICROSCOPY OF MIRROR SURFACES

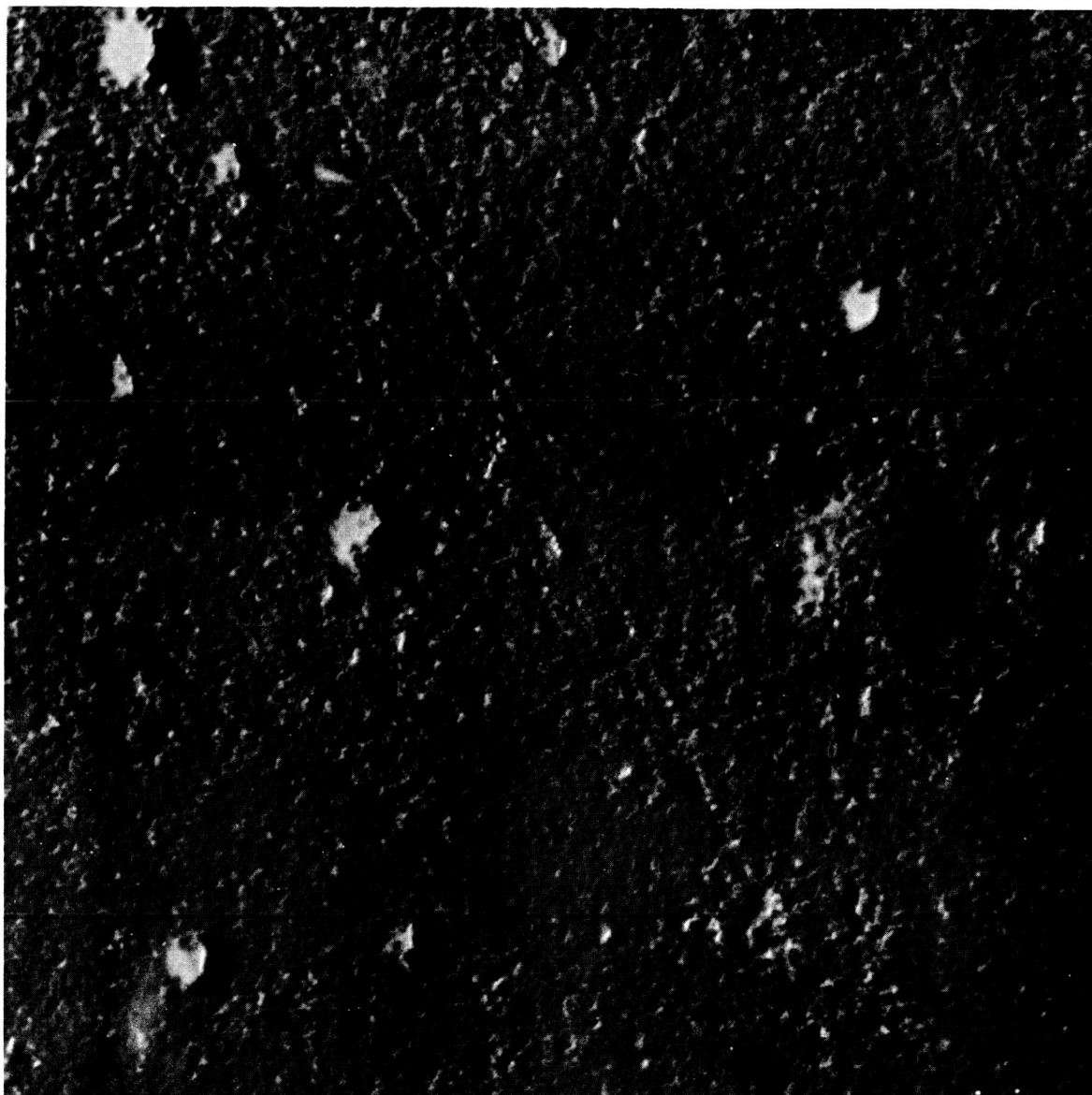
### 1. Specimen Film Material Experiments

For this set of three experiments, the shadow angle was standardized at  $9^\circ$  grazing and the shadow material was standardized as Pt-C. The three materials used were:

- 1) Thick Al + Mg-flash, later replaced by a C-film.
- 2) PVA (polyvinyl alcohol), later replaced by a C-film.
- 3) 1% Collodion in amylacetate, later replaced by a C-film.

The results of these experiments appear as Figs. 3 - 5.

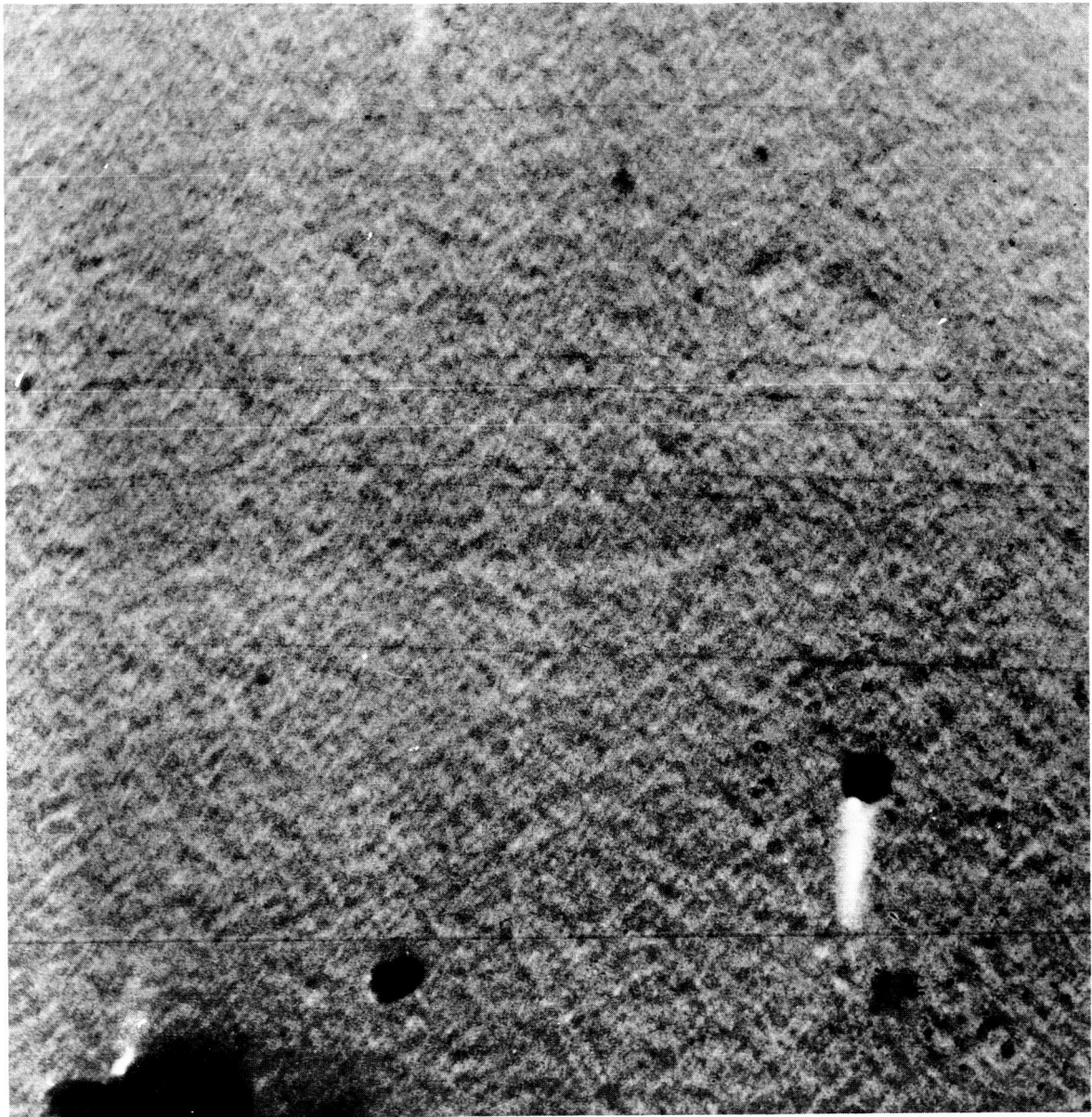
The Al + Mg-flash gives a replica with a multitude of detail. This set of experiments was carried out to try to be sure that any advantages of organic replication were not being overlooked. Also, exemplary specimens were desired for the record. We may not have evaded the prejudice in favor of the Pt-C that was caused by the continual quantitative systematic errors of about 50% that we found in grating groove step height using these organic films.



1μ

Fig. 3. Electron-micrograph of transfer Al film stripped from the surface of a microscope slide. The Al was removed in HCl after shadowing and after depositing C at normal incidence. Photographic Negative 212-6.





1μ

Fig. 4. Electron-micrograph of transfer PVA film surface from microscope slide surface. The PVA was removed in water after shadowing and after depositing C at normal incidence. Photographic Negative 200-28.



1 $\mu$

Fig. 5. Electron-micrograph of collodion film stripped from microscope slide. The collodion was removed in amyl acetate after shadowing and after depositing C at normal incidence. Photographic Negative 218-5.

## 2. Shadow Obliquity Angle Experiments

The shadow angle used for the grating and mirror electron-micrographs throughout the previous work on NONR 4277 (00)(X) was about  $19.7^\circ$  grazing.

In this experiment, the standardizations reported in Part I were used. In particular, the residual roughness of microscope slide surfaces as replicated by an Al-Mg film provided the shadow obstacles.

The unexpected result of this experiment was that a shadow angle of  $10.4^\circ$  grazing produced specimens, Fig. 6, whose image contrast was hardly any higher than the ones produced by  $19.7^\circ$  and  $32.4^\circ$  shadowing, Figs. 7 and 8. This is not the experience remembered for surfaces with more prominent obstacles.

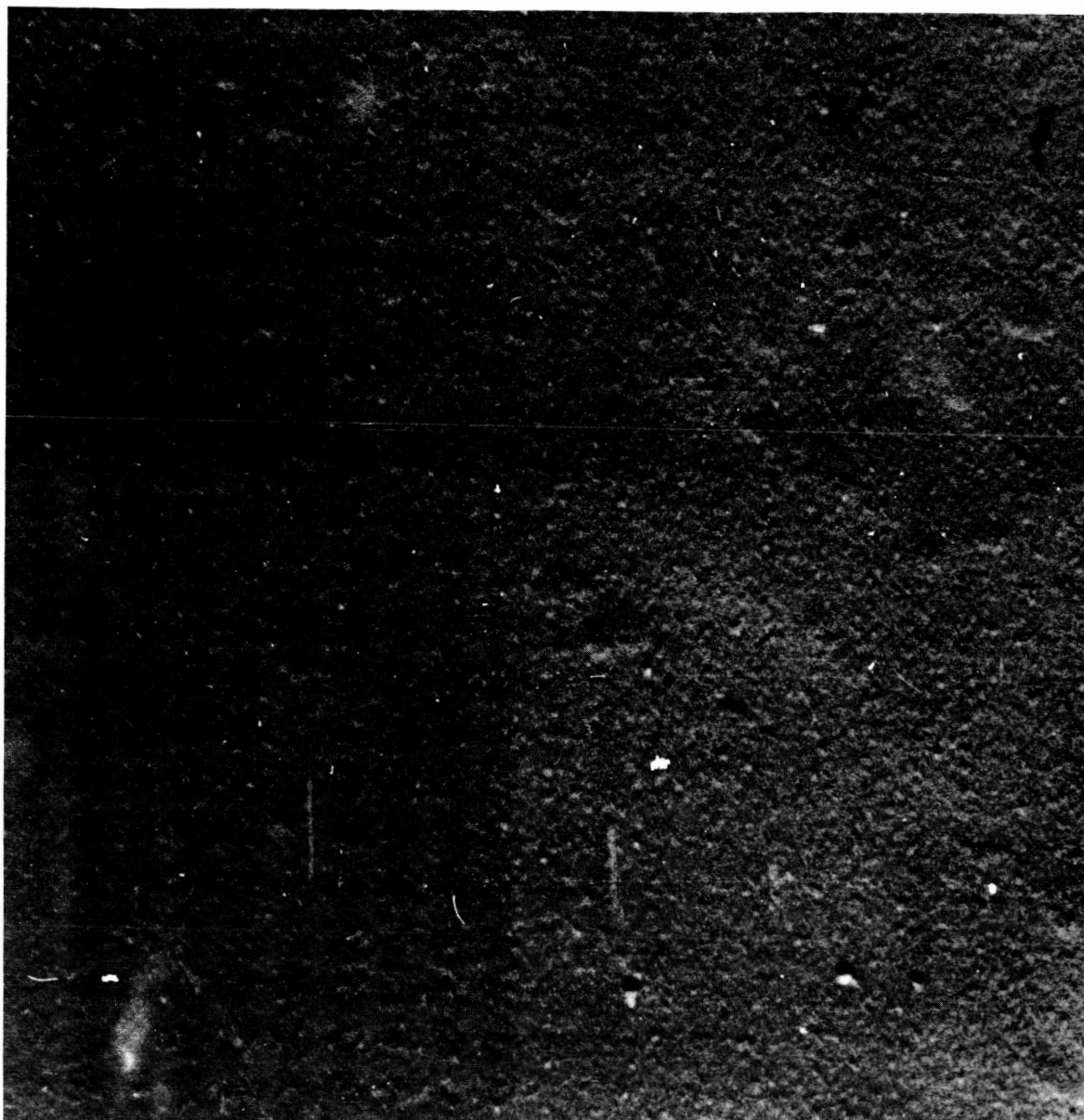
There are two physical effects that tend to reduce the contrast as the shadow angle approaches grazing. First, the evaporant source has height and therefore forms a penumbra zone whose width increases as the grazing angle decreases. Second, the roughness of deposited films is known to increase as the grazing angle decreases, particularly, from about  $40^\circ$  toward  $0^\circ$ .





1μ

Fig. 6. Electron-micrograph of an Al surface transferred from a smooth microscope slide. The grazing angle of the shadowing Pt-C film was 32.4 degrees. Photographic Negative 192-23.



1 $\mu$

Fig. 7. Electron-micrograph of an Al surface transferred from a smooth microscope slide. The grazing angle of the shadowing Pt-C film was 19.7 degrees. Photographic Negative 192-14.



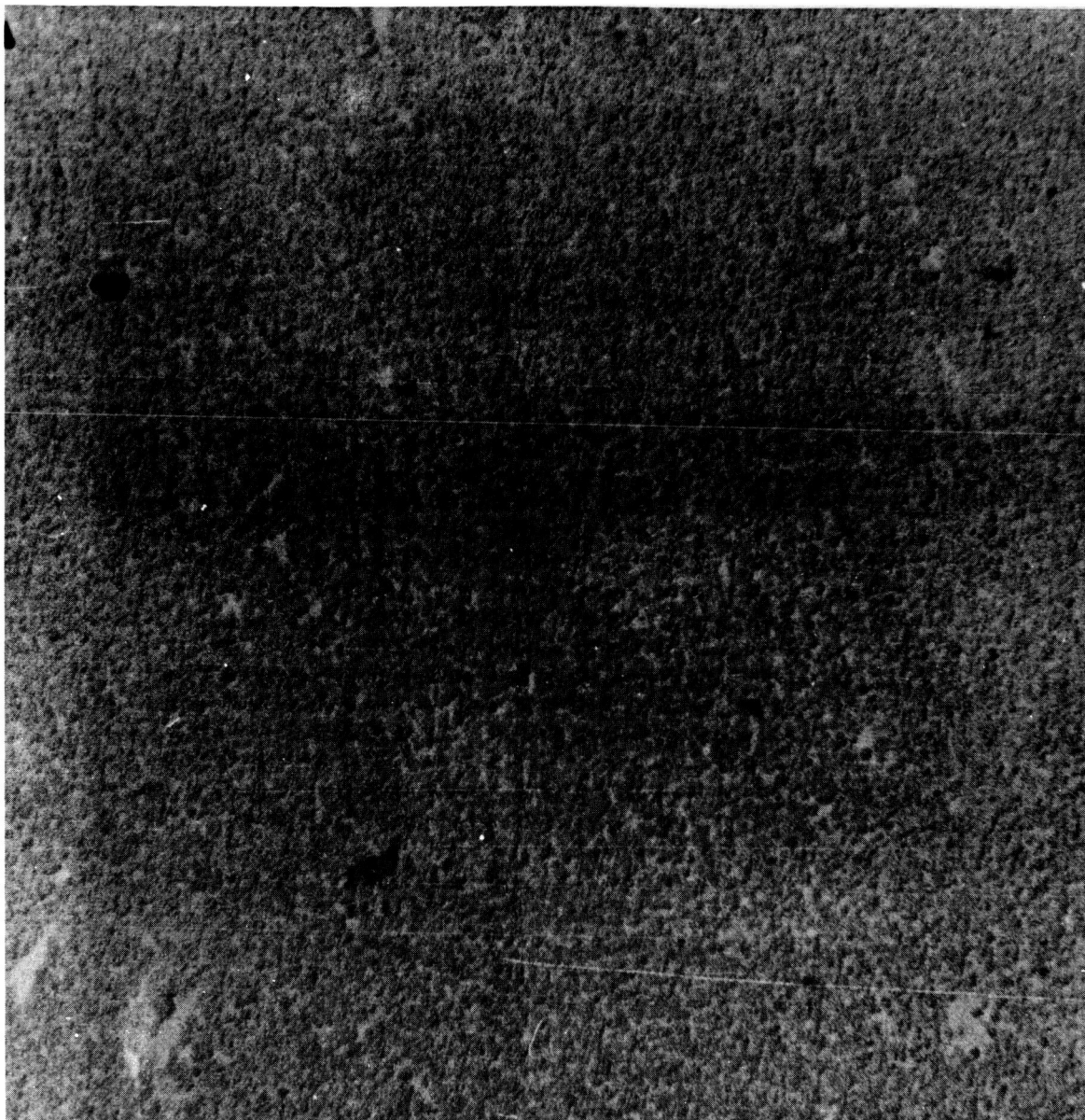
1μ

Fig. 8. Electron-micrograph of an Al surface transferred from a smooth microscope slide. The grazing angle of the shadowing Pt-C film was 10.4 degrees. The Pt together perhaps with some C is distilled from a carbon pellet by the heat of an electric current passing through the pellet. Photographic Negative 192-6.

### 3. Shadow Film Material Experiments

A report by Price of Dow Chemical Co. came to our attention after the work reported in this section was done. Our results fully confirm his report of October 27, 1964. Very recently, the use of uranium oxide shadowing has been suggested.

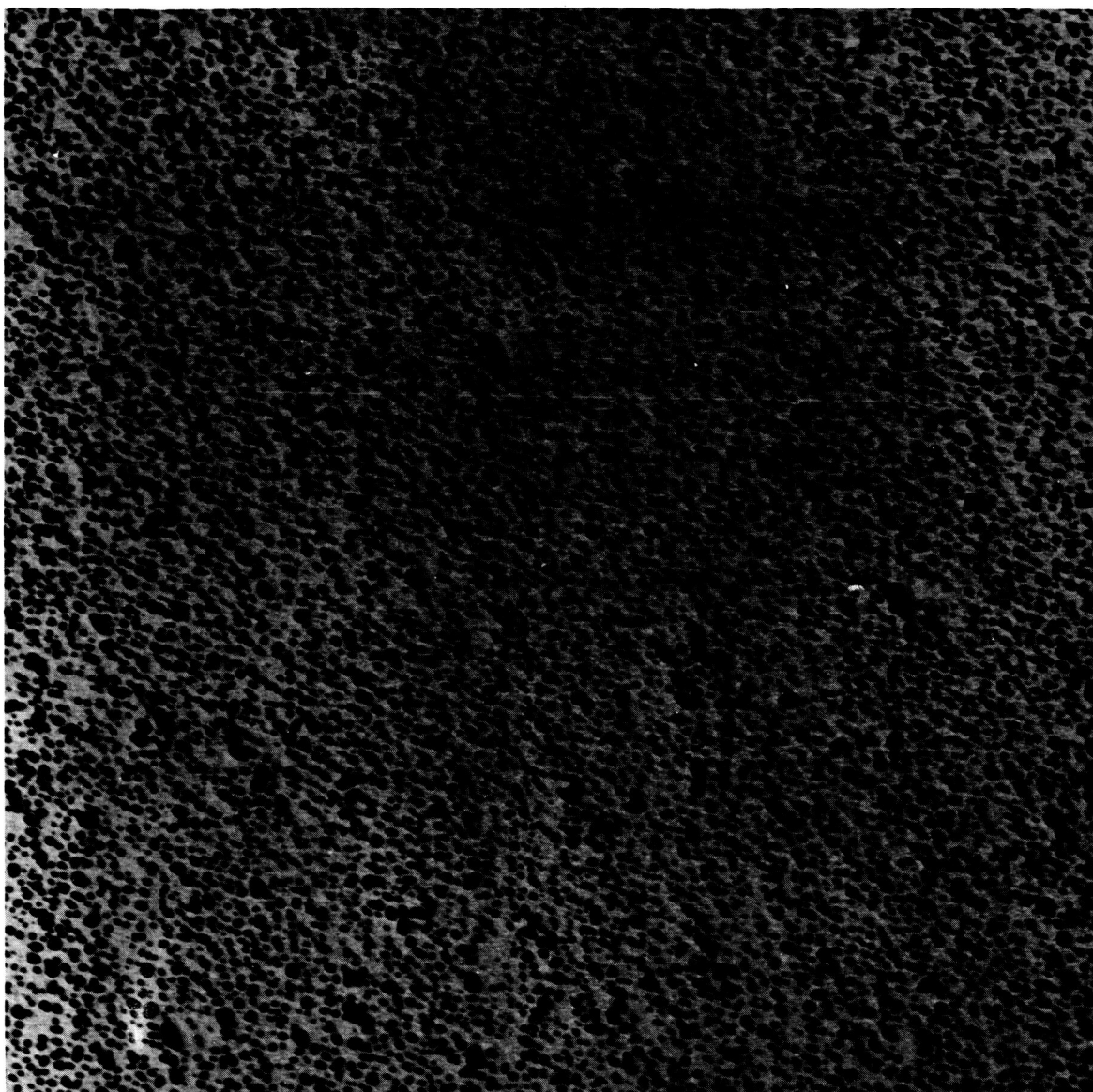
The following shadowing materials were deposited at about 9° grazing: 1) Pt-C, 2) Pd, 3) Au-Thermal Evaporation, 4) Au-Electron Beam Evaporation. Electron-micrographs of these appear in this report as Figs. 8 - 11.



1 $\mu$

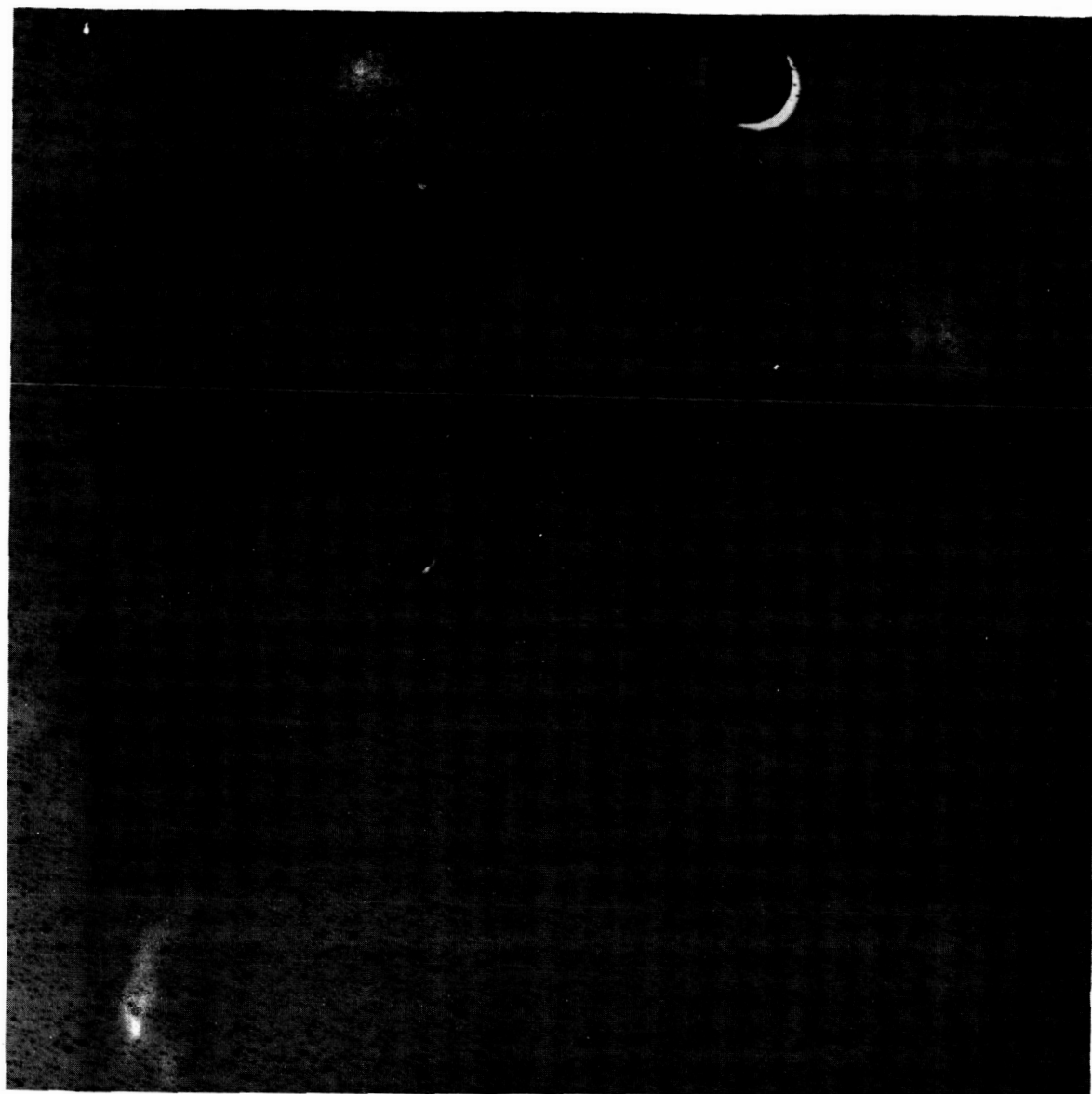
Fig. 9. Electron-micrograph of Al film transferred from a microscope slide then shadowed with Pd. The Pd is melted by current passing through a W wire around which the Pd wire was wrapped. Photographic Negative 201-6.





—  $1\mu$  —

Fig. 10. Electron-micrograph of Al film transferred from a microscope slide then shadowed with Au. The Au was held in a W wire "bird's nest" basket. The evaporation time was about 2 seconds. Photographic Negative 217-24.



Au

Fig. 11. Electron-micrograph of Al film transferred from a microscope slide then shadowed with Au. The Au was evaporated by electron gun heating. The time for shadow deposition was about 30 seconds. Photographic Negative 217-23.

## PART II SURFACE POLISH EFFECTS

There has been much opinion about the significance of surface polish for scattering losses in reflectance at short wavelengths.

This Part II describes observations of the smoothness of various substrates and observations both of the smoothness and specular reflectance of two types of mirror films, first surface and transfer, using various substrate qualities.

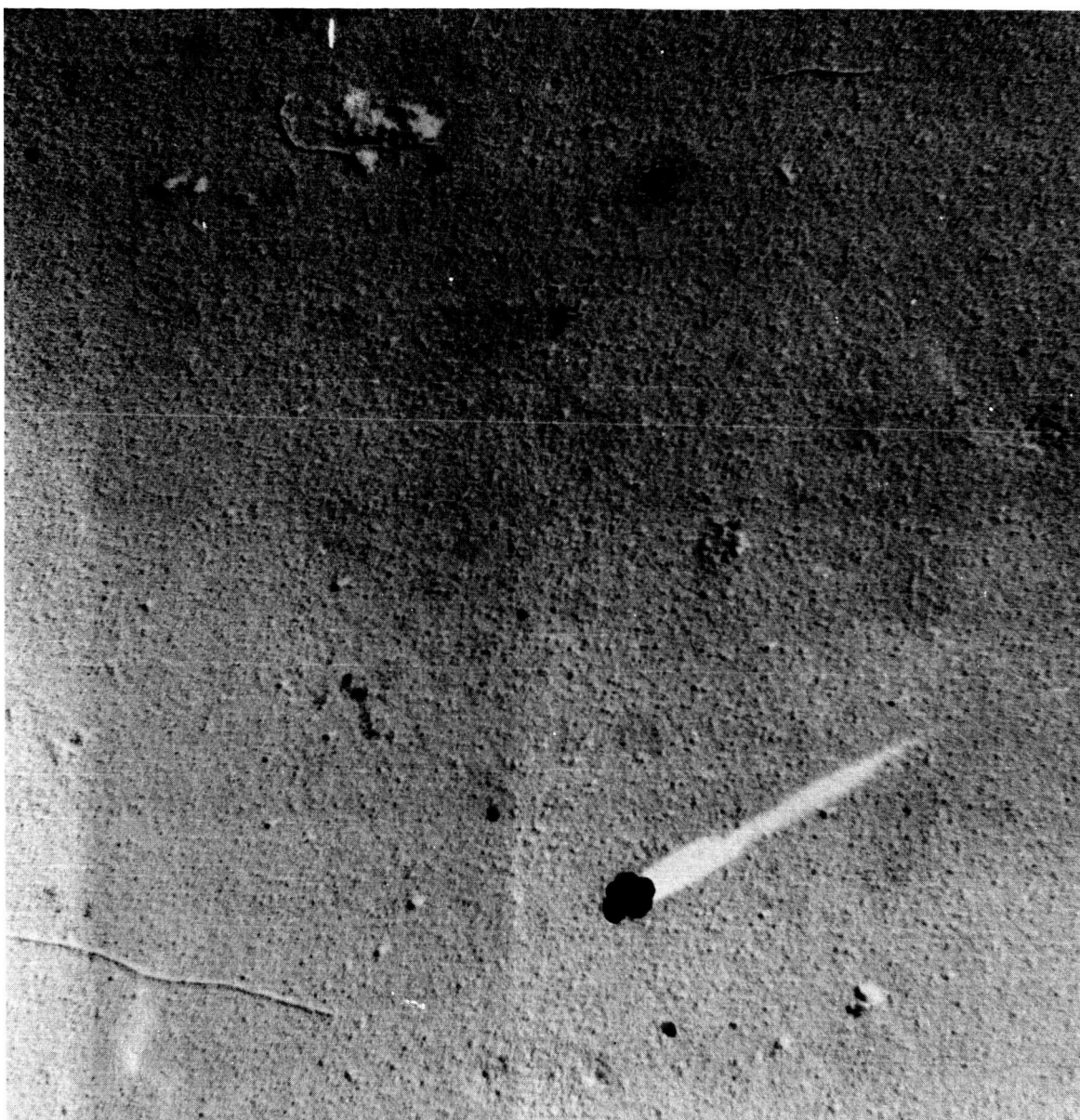
The electron microscope practices used were the ones reported in Part I. of this report. Transfer films of Al-Mg were prepared of each sample; they were shadowed at  $9^\circ$  grazing with Pt-C and coated normally with C; they were skived from the transfer substrate; the Mg and Al was dissolved; and the residual films were lifted out on Cu specimen screens.

Reflectance measurements were made at NRL using Mr. William Hunter's vacuum ultraviolet monochromator-spectrometer system as a reflectometer. The source was the pulsed d.c. source described in the Proceedings of the Xth Colloquium Spectroscopicum Internationale by W. R. Hunter.



## 1. Electron-Micrographs of Uncoated Surfaces

Figs. 12-18 show the residual defects on various surfaces; namely, floated collodion, cleaved mica, microscope slide, under-liquid-polished glass, optically-polished glass, buffed glass with light orange peel, buffed glass with heavy orange peel.



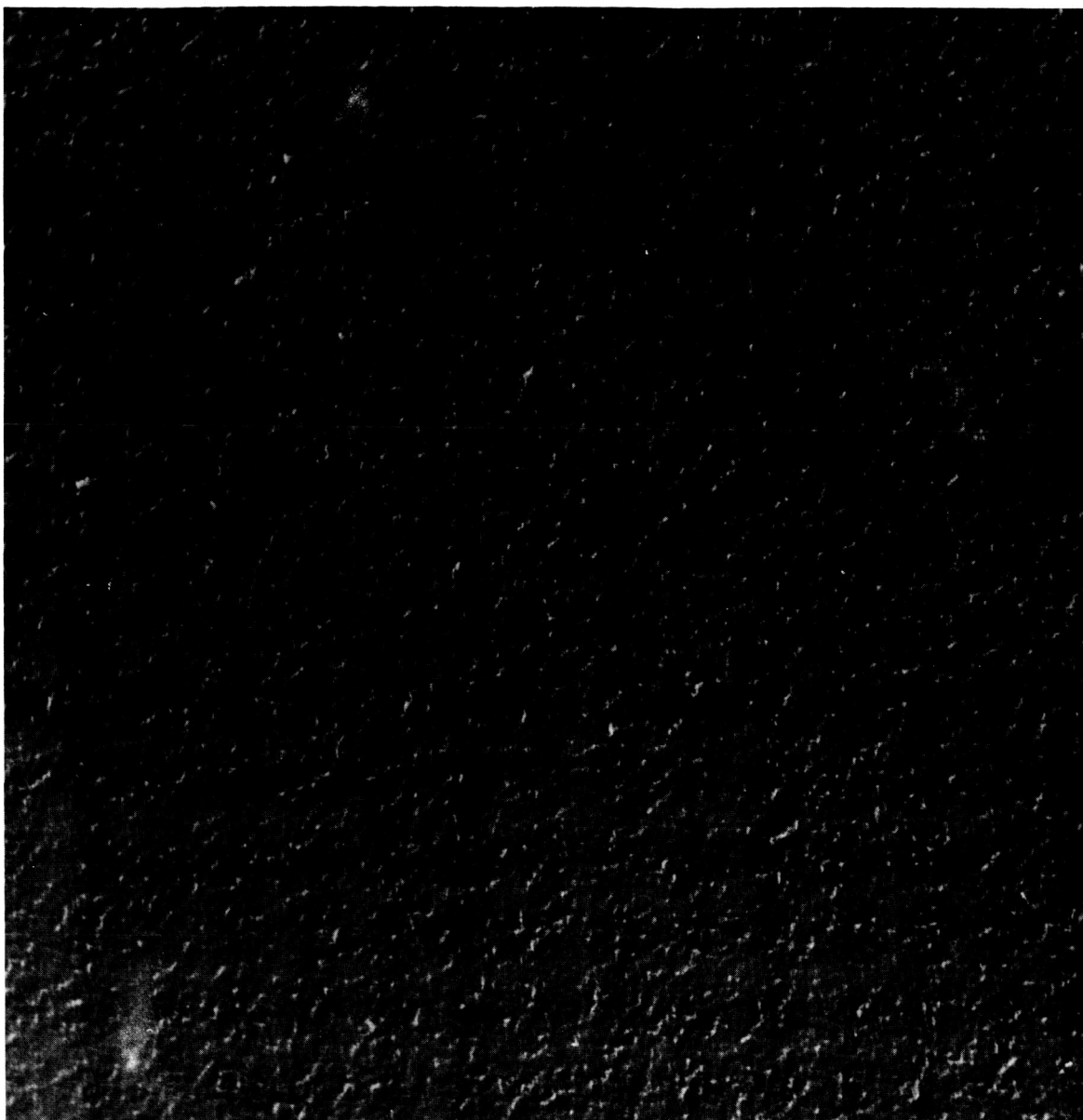
1μ

Fig. 12. Electron-micrograph made from a collodion film prepared by floatation on water of a 1% solution of collodion in amyl acetate. Photographic Negative 218-23.



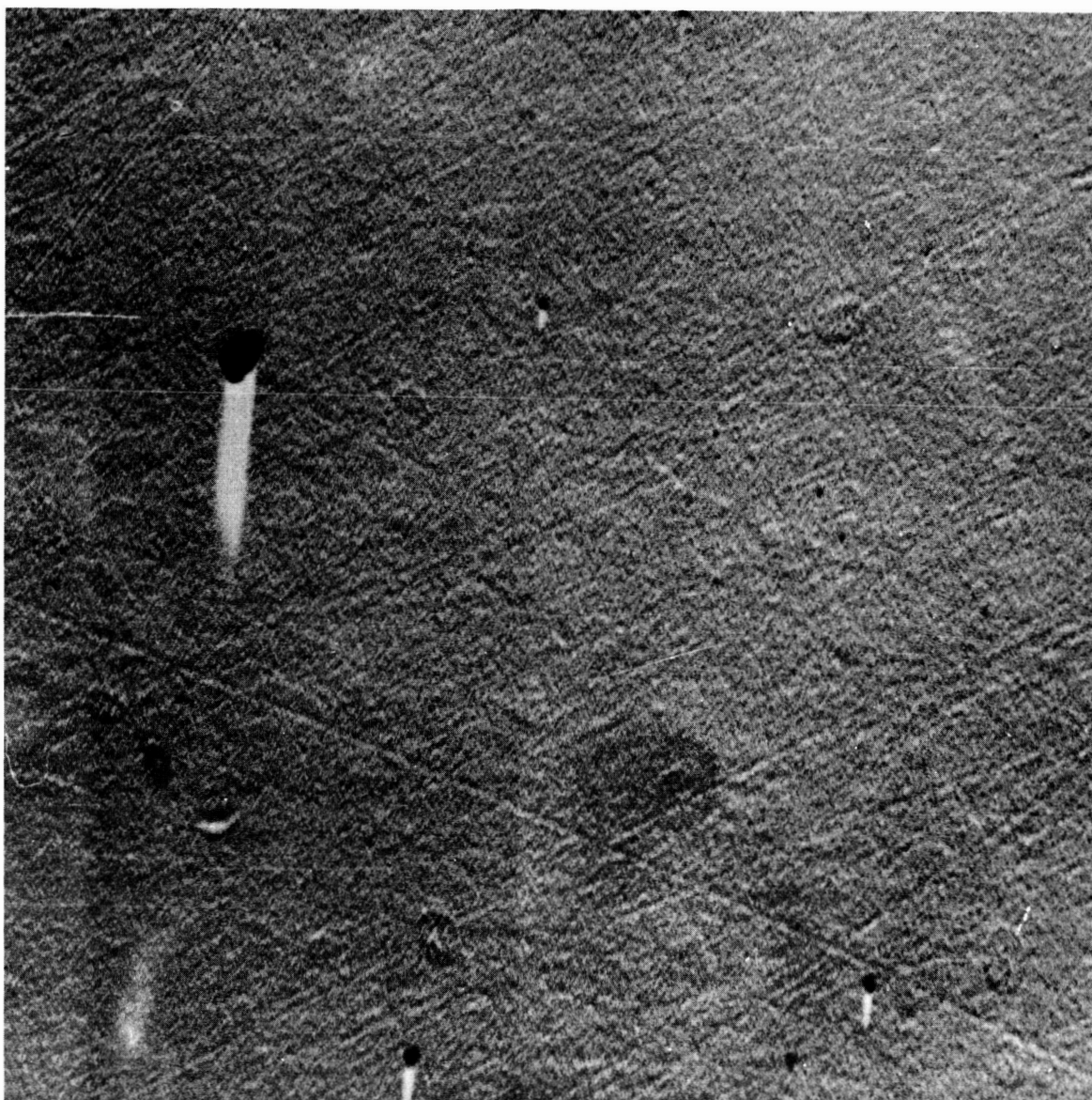
1μ

Fig. 13. Electron-micrograph of the surface of  
cleaved mica. Photographic Negative 201-21.



1μ

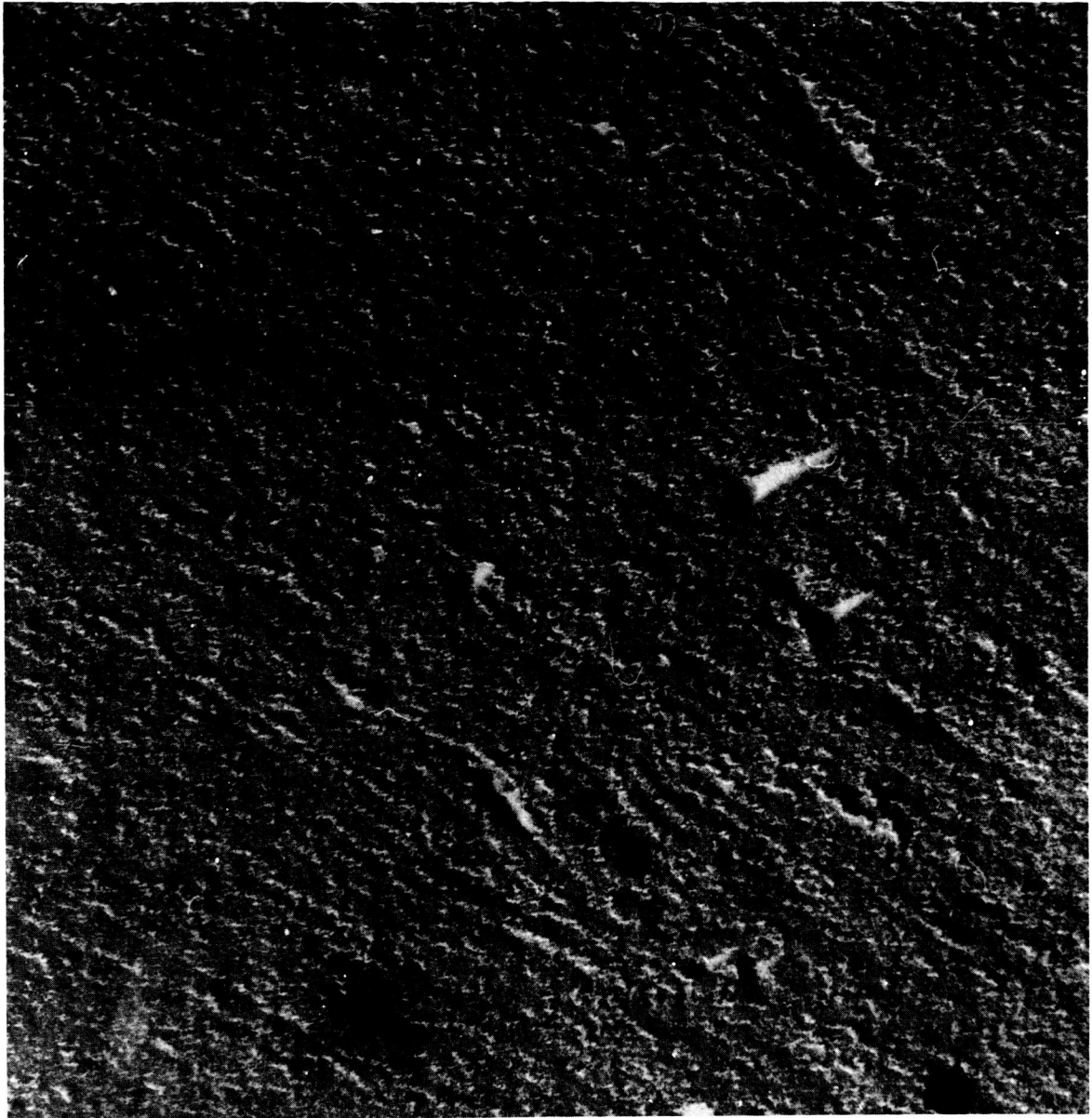
Fig. 14. Electron-micrograph of the surface of a microscope slide, nominally fire polished. Photographic Negative 192-6.



1μ

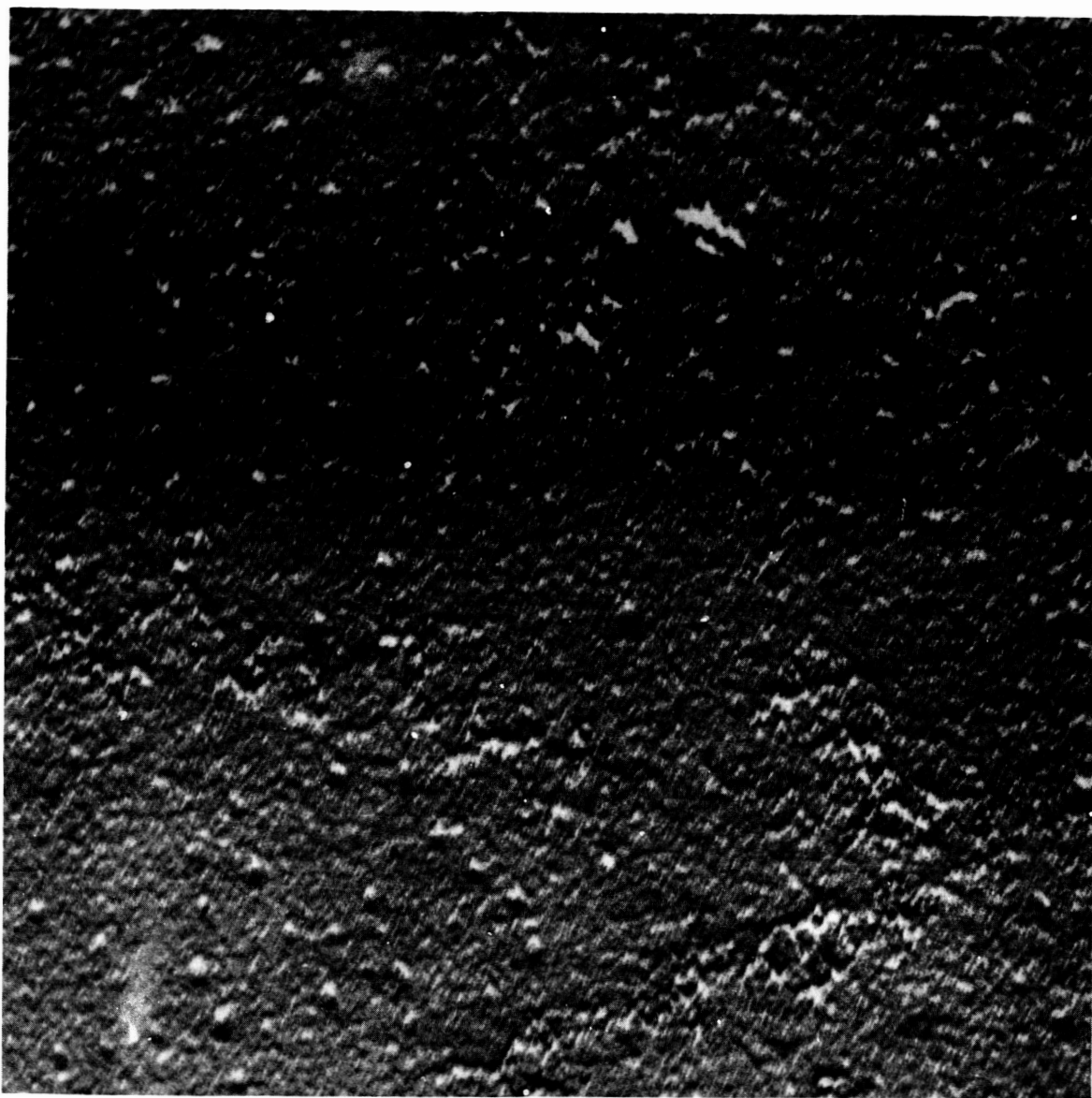
Fig. 15. Electron-micrograph of the surface of glass polished-under-liquid. Photographic Negative 212-24.





1 $\mu$

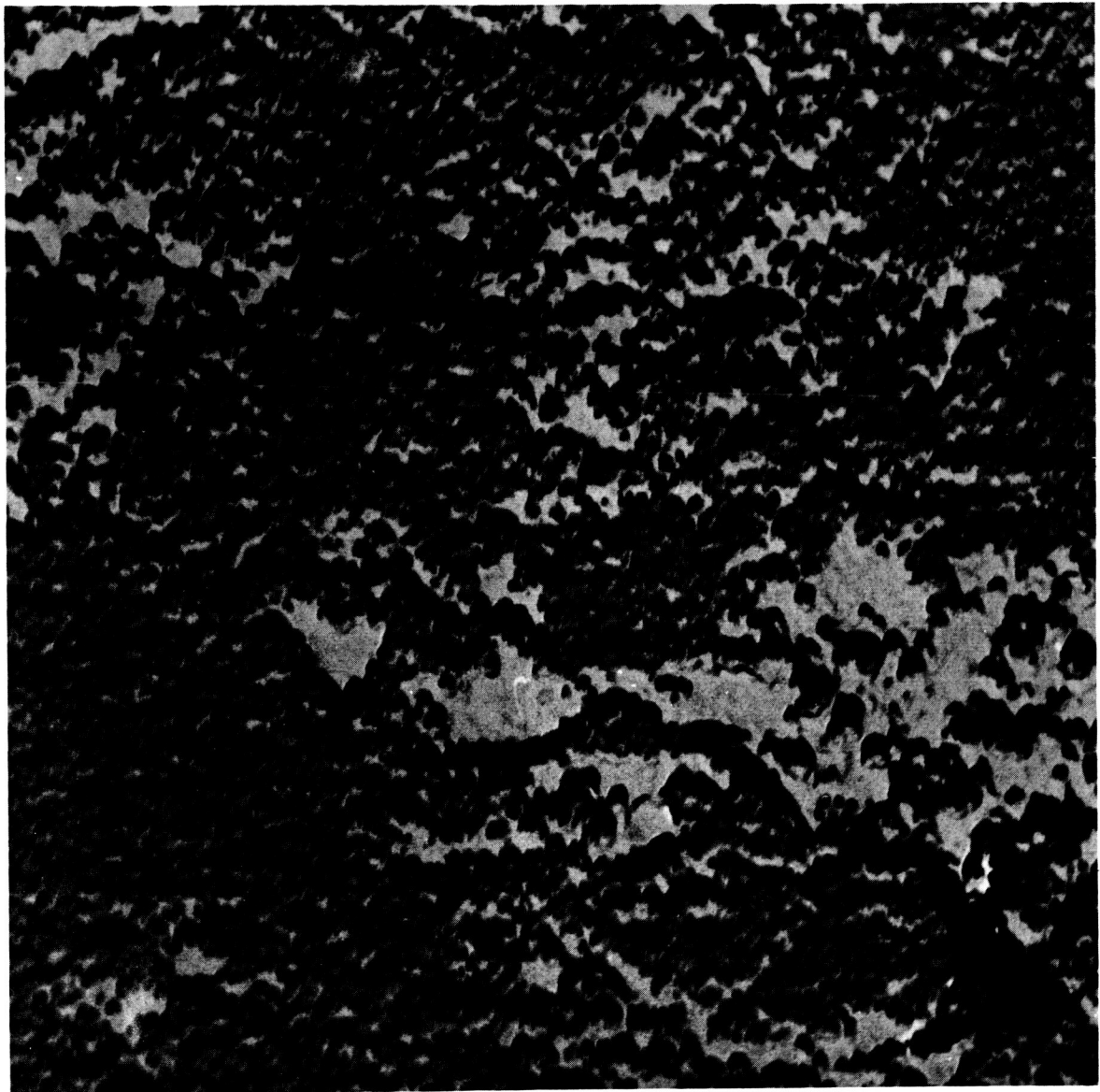
Fig. 16. Electron-micrograph of the surface of glass polished to an optical finish, free of defects at 8 power. Photographic Negative 199-4.



1μ

Fig. 17. Electron-micrograph of the surface of glass with a slight orange-peel acquired by buffing.

Photographic Negative 212-7.



1μ

Fig. 18. Electron-micrograph of the surface of glass with a severe orange-peel finish. Photographic Negative 212-10.



## 2. Electron-Micrographs and XUV Reflectances of First Surface $.4\mu$ Au Films

The gold thickness of  $.4\mu$ , used here for the comparisons of substrate influence, corresponds to the thickness that is often used for a master grating ruling. The films on the four following electron-micrographs, Figs. 19-22, were deposited simultaneously on glass with various surface finishes. The deposition was performed by heating the gold with an electron gun. The average deposition rate was  $10.3\text{A/sec}$ .

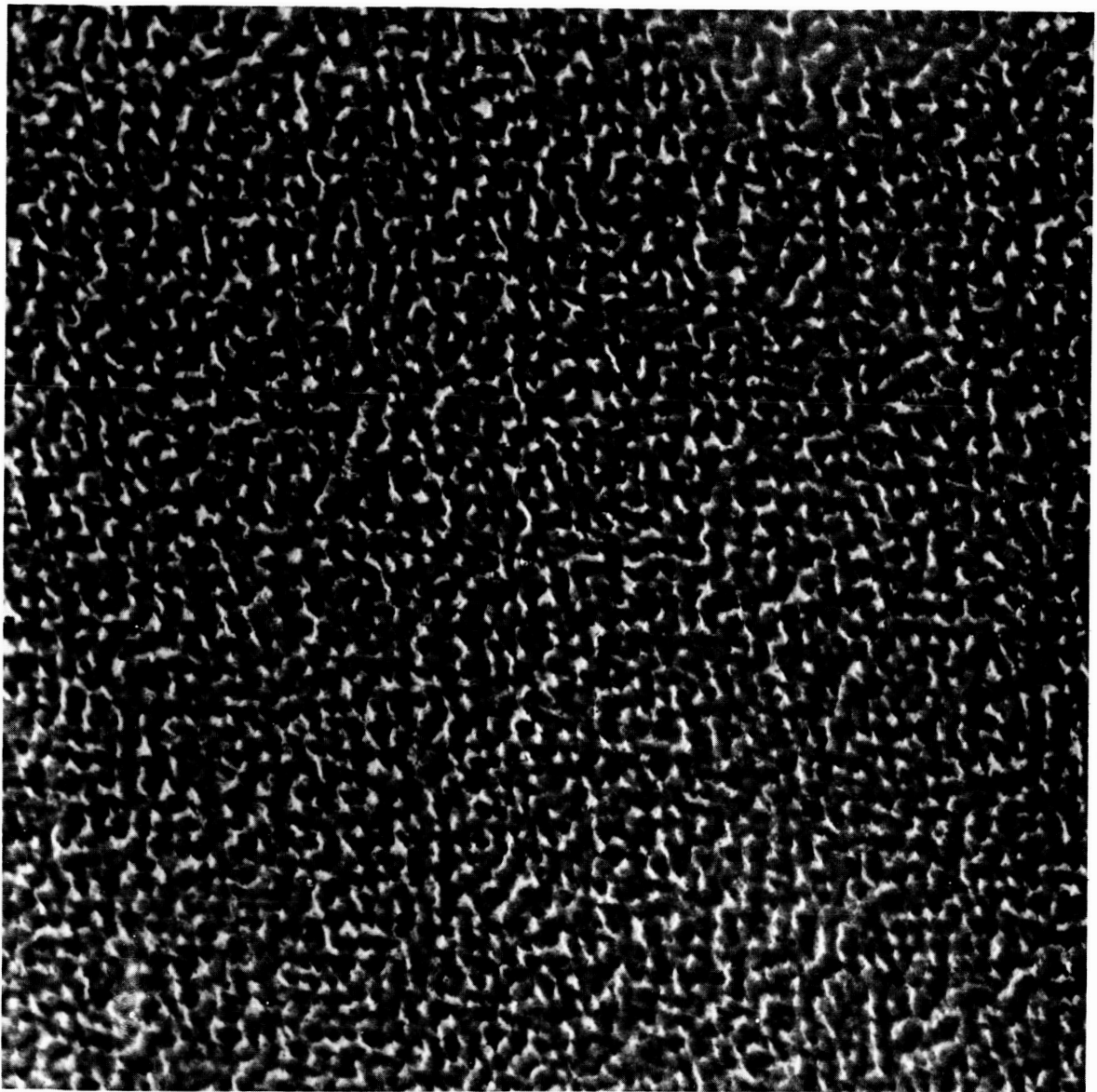
There is little difference in the appearance of the electron-micrographs despite the difference in the substrates.

The reflectances in Table I show that there is no difference significantly larger than the measured 0.5% average systematic and 0.5% average RMS difference found for a repeated setup and measurement of the same mirror.

Table I. Comparative Reflectance of Simultaneously-Deposited  
Au Films on Substrates with Different Polish.

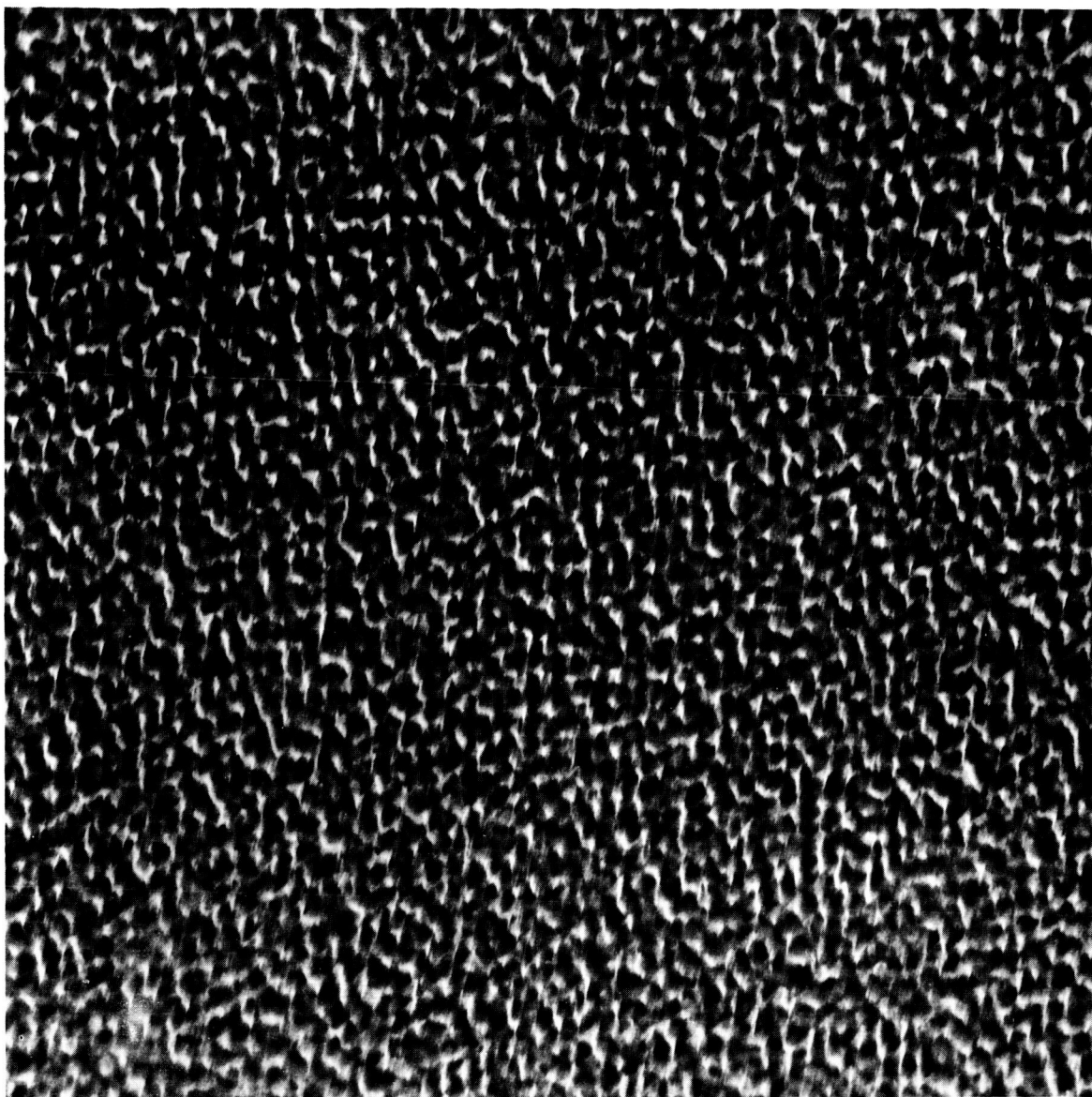
| <u><math>\lambda</math><br/>in Å</u> | <u>Under-<br/>liquid</u> | <u>Micro-<br/>slide</u> | <u>Optical<br/>Polish</u> | <u>Buffed</u> |
|--------------------------------------|--------------------------|-------------------------|---------------------------|---------------|
| 304                                  | .1                       | .19                     | .1                        | .1            |
| 463                                  | .4                       | .5                      | .9                        | .6            |
| 508                                  | 1.2                      | 1.2                     | .9                        | 1.4           |
| 554                                  | 2.5                      | 2.7                     | 2.4                       | 2.5           |
| 610                                  | 3.3                      | 3.6                     | 3.0                       | 3.3           |
| 686                                  | 3.0                      | 3.4                     | 2.9                       | 3.0           |
| 788                                  | 4.2                      | 4.6                     | 4.1                       | 4.3           |
| 899                                  | 6.4                      | 6.5                     | 6.6                       | 6.8           |
| 1085                                 | 6.0                      | 6.3                     | 6.4                       | 6.6           |

The RMS difference is approximately 0.22 for the readings in the above table, somewhat less than repeated measurements made on one mirror.



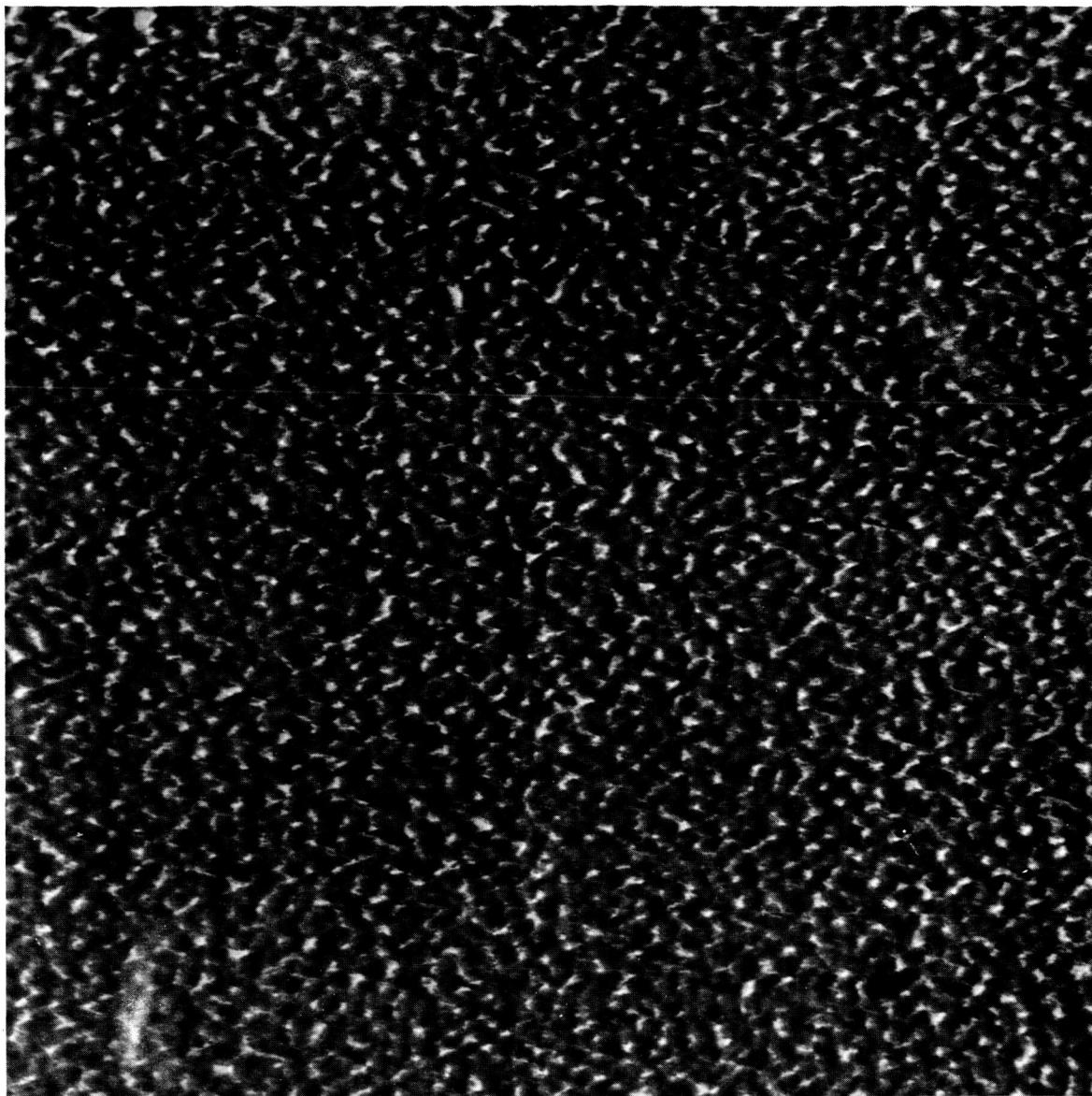
1μ

Fig. 19. Electron-micrograph of a first surface gold film  
on a Cr flashed microscope slide. Photographic Negative 216-15.



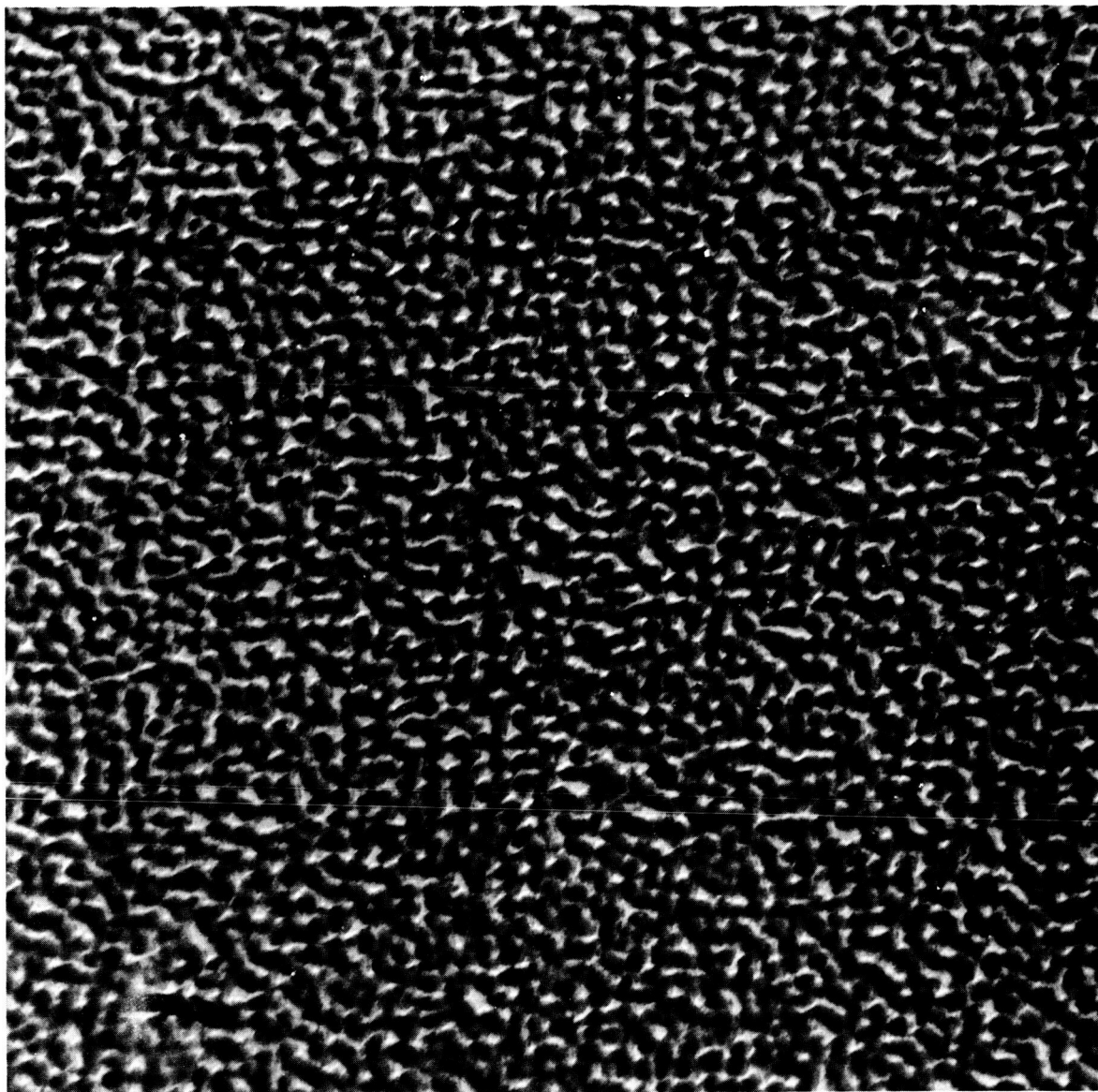
1μ

Fig. 20. Electron-micrograph of a first surface gold film  
on Cr-flashed glass that had been polished under liquid.  
Photographic Negative 216-8.



1 $\mu$

Fig. 21. Electron-micrograph of a first surface gold film on Cr-flashed glass that had been conventionally polished to optical quality, free of defects observable with 8 power magnification. Photographic Negative 216-5.



1 $\mu$

Fig. 22. Electron-micrograph of a first surface gold film on Cr-flashed glass that had been buff-polished to produce an orange-peel finish. Photographic Negative 216-20.

### 3. Electron-micrographs and XUV Reflectances of Au(Cr) Films Transferred from Surfaces of Differing Polish.

The electron micrographs, Figs. 23-26, associated with this section are of samples prepared simultaneously as follows:

- a. About .4 microns of Au was deposited in about 7 minutes simultaneously onto four different glass polishes (microscope slide, under-liquid polished glass, optically-polished glass, and buff-polished or shined glass).
- b. Less than 0.1 micron of Cr was deposited on the Au.
- c. The exposed Cr surface was cemented to a microscope slide with epoxy.
- d. After curing, the glass plates were separated at the Au-to-glass interface.
- e. A standard Al film transfer specimen of this exposed Au surface was then made, shadowed at  $9^\circ$  with Pt-C, and converted into a specimen for electron microscopy as described previously in this report.

The reflectance of the transferred gold films is reported in Table II.

The glass that was polished under liquid produced a transfer mirror that at every wavelength was observed to have higher

reflectance than any of the other three. It averaged more than 2% absolute (about 30% relative) higher than any of the other types.

A remeasurement of the mirror transferred from optically-polished glass showed a systematic measurement difference of 0.5% and an 0.5% random RMS difference from the corrected average. Thus, any one measurement is quite likely wrong by 2%, and any one set of measurements is quite likely systematically wrong by 1%. This range of error is entirely consistent with simple reflectometer experience in other parts of the spectrum. Unfortunately, it masks the subtle differences that we would like to be sure about.

The higher reflectance of the mirror transferred from the glass polished under liquid is believed to be a real effect.

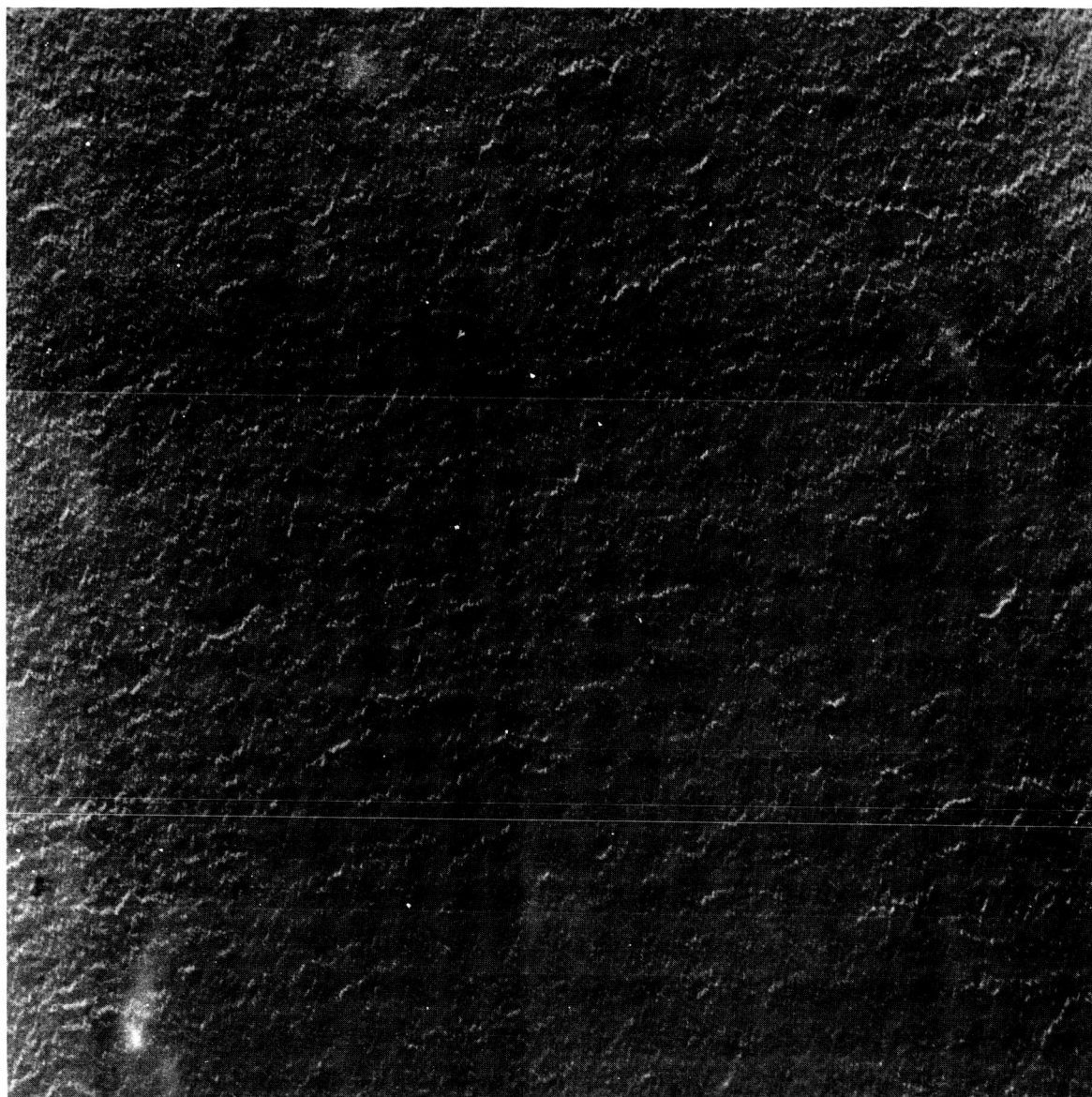
The mirror that was transferred from a buffed glass plate had a 0.3% lower average reflectance than the average of the micro-slide and optically-polished transfer mirrors. This is not a significant difference, and even if real, it is a small difference in view of the micrograph appearance.



Table II. Comparative reflectance of Au films simultaneously deposited on substrates with different polish, then transferred to expose the Au-to-glass interface.

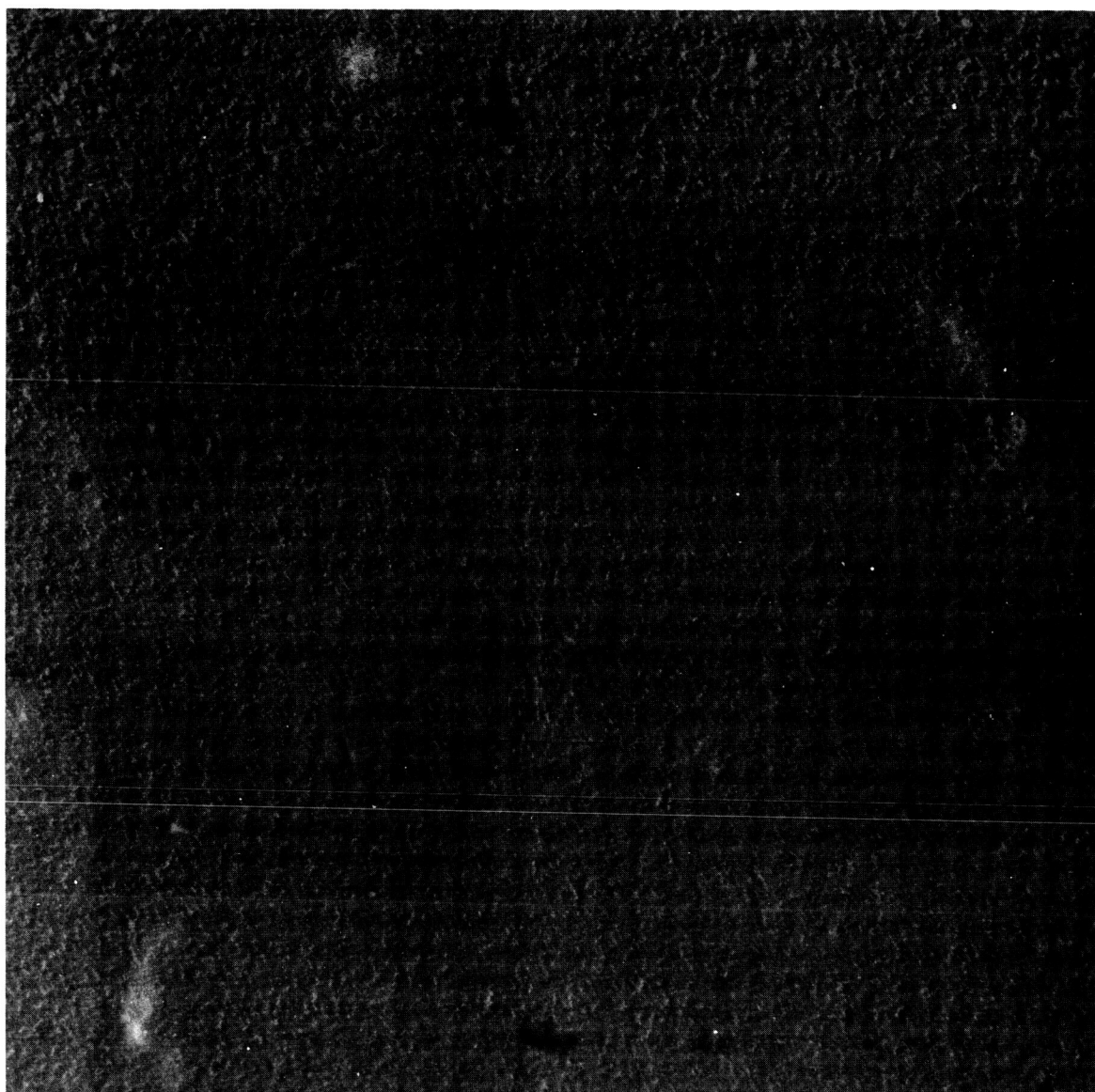
| $\lambda$<br>A | Bowl<br>Pol. | Micro<br>-Slide | Opt.<br>Pol. | Buff<br>Pol. | C.H.&H. | $\lambda^{-1}$<br>kk |
|----------------|--------------|-----------------|--------------|--------------|---------|----------------------|
| 283            | 2.8          | 1.8             | 1.8          | 1.7          | 3.3     | 353                  |
| 304            | 3.3          | 2.7             | 2.6          | 3.3          | 3.0     | 333                  |
| 322            | 4.1          | 2.9             | 2.9          | 2.8          | 3.7     | 311                  |
| 345            | 4.9          | 4.0             | 3.4          | 3.7          | 5.0     | 290                  |
| 374            | > 7.2        | > 5.4           | > 5.2        | > 5.2        | 8.0     | 267                  |
| 419            | > 7.2        | > 6.2           | 6.9          | > 5.4        | 7.5     | 239                  |
| 462            | > 8.1        | > 6.9           | 8.1          | > 6.1        | 7.5     | 216                  |
| 508            | 14.5         | 11.7            | 9.6          | 9.9          | 12.0    | 197                  |
| 554            | 18.5         | 15.2            | 15.7         | 14.7         | 16.5    | 181                  |
| 609            | 15.7         | 13.7            | 14.3         | 11.6         | 12.5    | 164                  |
| 686            | 11.2         | 10.8            | 10.7         | 8.9          | 9.6     | 146                  |
| 787            | 13.9         | 11.6            | 10.7         | 10.5         | 11.5    | 127                  |
| 898            | 14.7         | 11.4            | 11.5         | 12.1         | 13.0    | 111                  |
| 1085           | 13.6         | 9.9             | 8.7          | 10.9         | 14.9    | 92                   |

The section of the data surrounded by a box was taken at 1X from the recorder readings which have here been increased by 0.7% in order to correct for the 0-point recorder error observed. The greater-than symbol appears ten times above because the direct beam was not corrected for background.



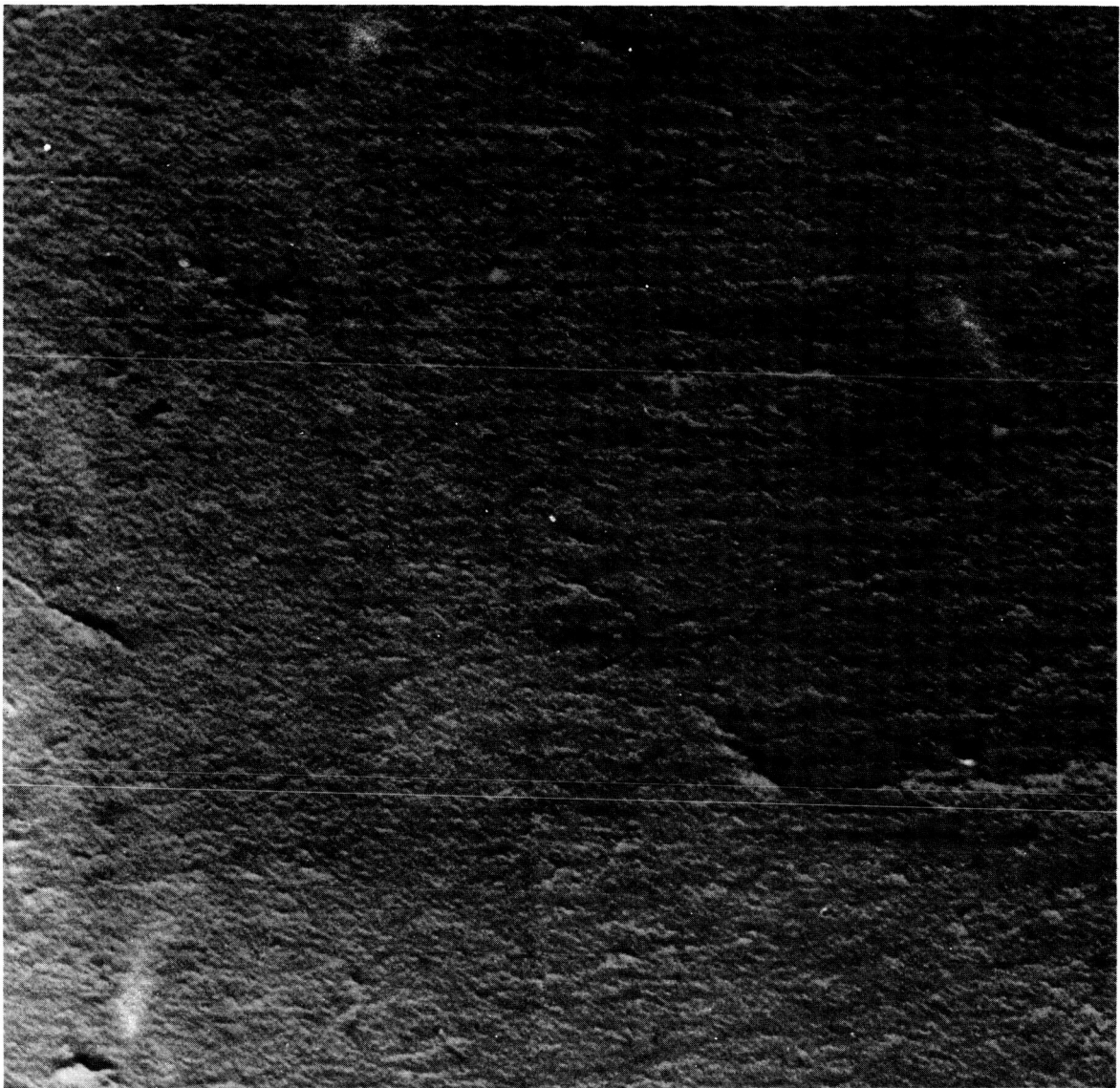
1 $\mu$

Fig. 23. Electron-micrograph of transferred Au surface  
originally deposited on a microscope slide.  
Photographic Negative 217-15.



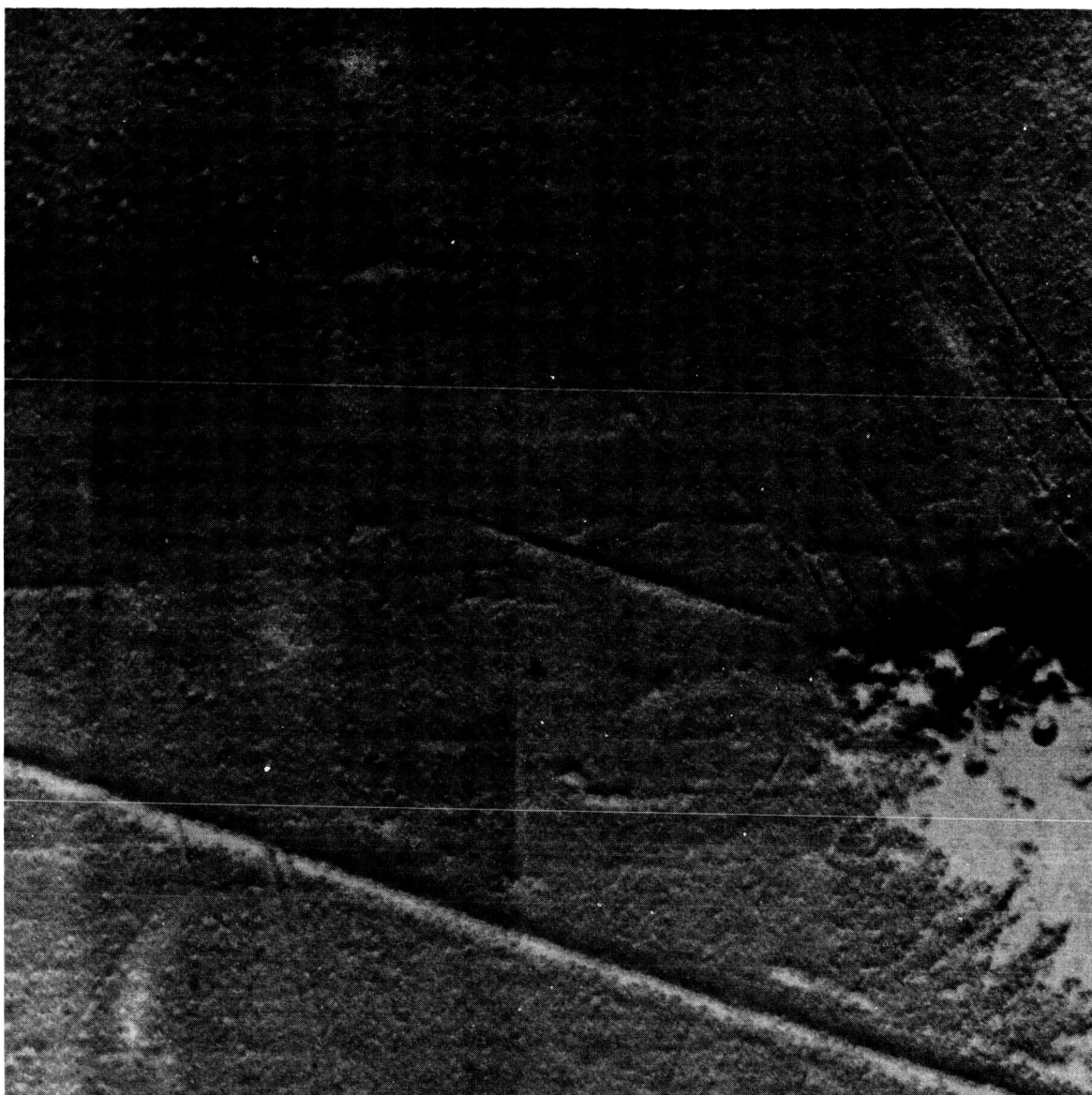
1 $\mu$

Fig. 24. Electron-micrograph of transferred Au surface originally deposited on a glass surface that had been polished under liquid. Photographic Negative 217-9.



1μ

Fig. 25. Electron-micrograph of transferred Au surface originally deposited on a glass surface that was free of defects at 8X. Photographic Negative 217-4.



1μ

Fig. 26. Electron-micrograph of transferred Au surface originally deposited on a glass surface that was polished by buffing. Photographic Negative 217-19.



#### 4. Reflectometer Accuracy

In the fourth quarterly report on Contract NONR 4277, dated Jan. 31, 1965, a section entitled, "Standard (10/23/64)" Curve, was included. It contained the highest reflectance values at each wavelength that had been measured during the grating study performed by B&L with NRL equipment. These high measurements were not confirmed by remeasurements at a later time but they were remeasured several times during the October measurement session.

Careful attention has been paid to possible sources of error due to the equipment and due to its operation. A few judgements seem worth making at this time.

Very large errors could result if the detector position in the optical beam varied from the direct beam to the reflected or diffracted beams. We believe that this was responsible for the excessively high values measured in October 1965.

A further type of misalignment was observed in July 1965 when Mr. Angel got different reflectances when he rotated the sample mirror from ones when he rotated the detector. This difference was noted when the detector was set at  $10^\circ$  ( $5^\circ$  incidence) but not when set at  $15^\circ$ . The possibility of some interference at some wavelengths is suggested by this observation plus the known slight variation of maximized directions with wavelength.

Every reading made at 924A in July was found to depart markedly from the best curve for all other wavelengths. The 924A line was exceptionally strong so that the photomultiplier may have been overloaded and non-linear.

Particular care in reading the background was found important for the reflectance measurements made early in September 1965. At the peak of the spectral reflectance curve of gold, near 554A, the background was uniformly zero for all measurements. Comparisons made at this wavelength do not therefore involve any background correction. At 304A however, the background ranged between 22 and 25 parts per hundred of the direct beam. The reflectance is selectively low at this wavelength; so the background contribution to the reflected signal is large and the measurements are open to much question.

Many reflectance measurements made in September 1965 were made in three ways: 1X on the recorder, 1X on the meter, and 10X on the recorder. Reflectances at 1X on the recorder were measured consistently lower than at 10X or at 1X on the meter. Differences as large as 0.7 between the 1X meter and 10X readings occurred for a small percentage of the readings but without a consistent winner, consequently they have been treated as equivalent - the 10X recorder readings are tabulated in Tables I and II. Previously, when measuring gratings, weak diffraction orders could often be clearly observed on the meter even when they could not be discovered with the recorder.



### PART III - FLASH-COATED TRANSFER MIRRORS

The expression, flash-coated, refers to a rapidly deposited thin film of metal. The purpose of the rapidness of the deposition is to obtain high optical reflectance which depends most importantly on deposition rate for whatever metal is selected.

We have suspected that flash-coatings have an especially high density of micro-lumps or spit. In extreme cases, the evaporating charge emits multitudes of projectiles large enough to be seen through the bell jar window. Large particles of this sort may contain  $10^{10}$  atoms, in round numbers. We believe that the distribution of emitted lump sizes may be random and therefore, Poisson in character. From the frequency of large lumps, this implies the average particle size to be much greater than monatomic. The anticipation that the size of the particles being evaporated is monatomic comes mainly from oven-evaporation experiments such as vapor absorption demonstrations and Stern-Gerlach results. The literature of metal film deposition accepts the monatomic distillation assumption so far as we know. This assumption is not valid for optical mirror deposition.

Theoretically, there is a difficulty in explaining how a vapor particle becomes bound to a substrate surface of a different material.

Experimentally, there is a difficulty with the idea of using a pre-deposited film surface as the substrate on which the flash-coating is deposited. Lumpiness apparent on flash-coatings is not distinguishable inherently from lumpiness present in the underlying film.

Transfer films avoid this difficulty because their surfaces are extremely smooth. Any lumps that appear come with the flash coating.

Furthermore, the surface of a gold film is perhaps less changed from its pure metallic condition than other metals.

Figs. 27-29 are electron-micrographs made by the procedures described in PART I. In each case, a gold transfer film was used. Fig. 27 is a picture of a gold transfer surface without any flash-coating. Figs. 28 and 29 have thin gold and platinum respectively deposited on the underlying gold transfer surface.

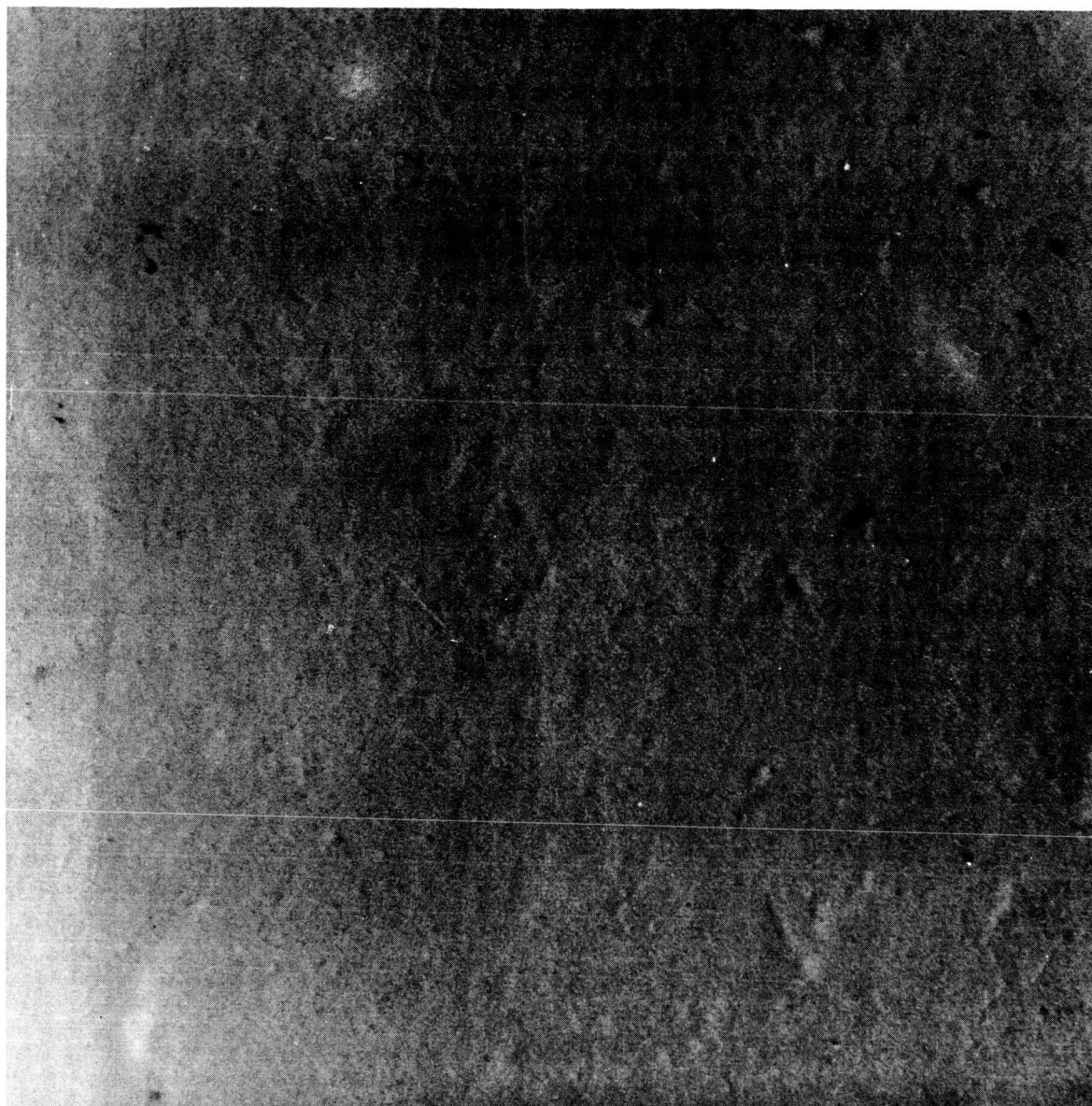
These thin over-coatings were made by electron gun evaporation from a pool of metal that was melted while the transfer film was protected by a shutter. The time of over-coating deposition was 13 sec. for the gold and 4.3 min. for the platinum. The electron gun available for this test could not be operated for more rapid Pt deposition without the risk of arcing to ground.

Despite the comparatively slow deposition rate, many nuggets of metal that struck the transfer film during the flash-coating appear in the approximately 25 square microns shown in Figs. 28 and 29.

Clear glass plates coated simultaneously with the flash-films were measured in the visible to have the following properties:

Au-flash:     $R = .59$         and         $T = .26$

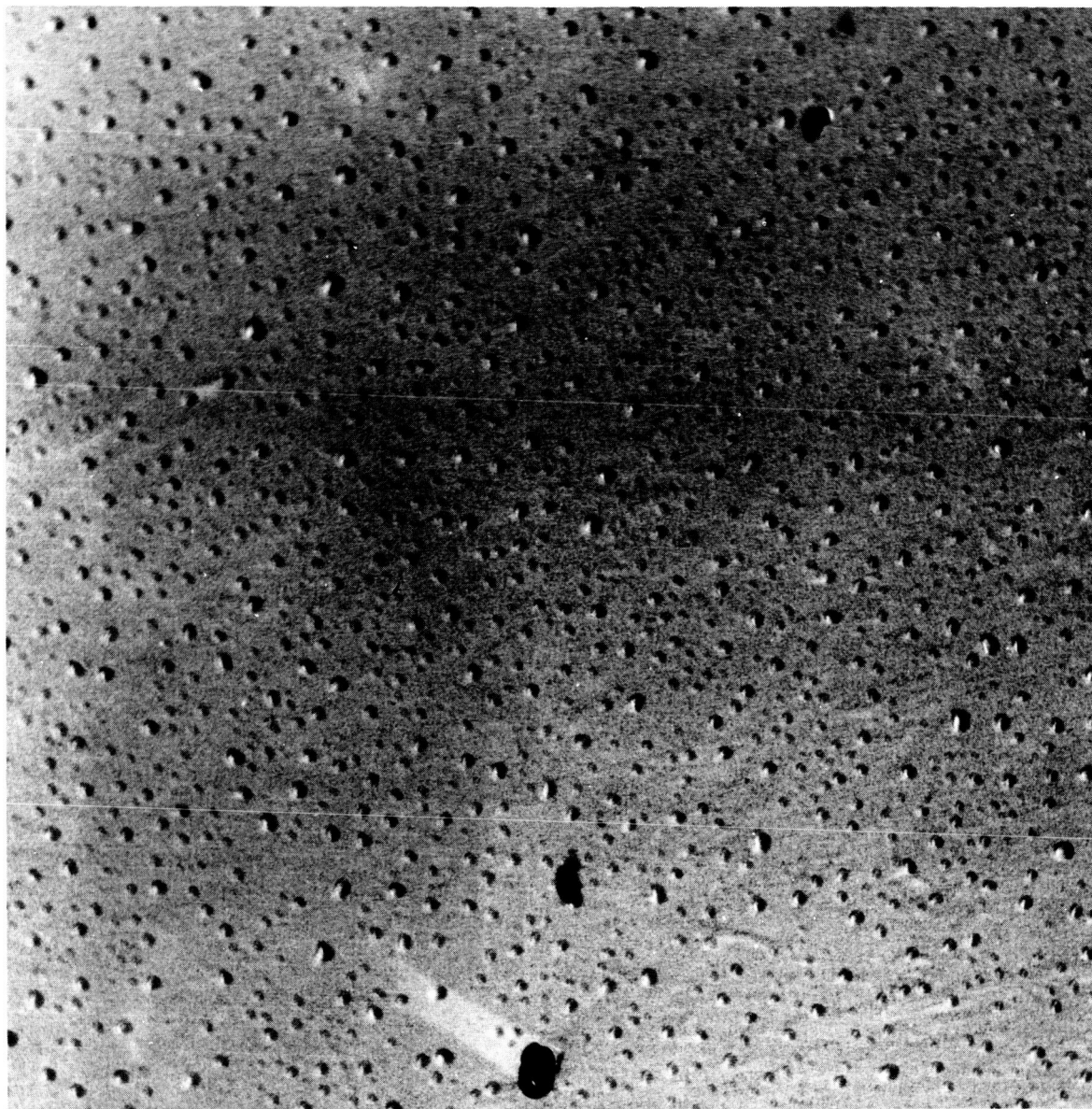
Pt-flash:     $R = .23$         and         $T = .44$



1 $\mu$

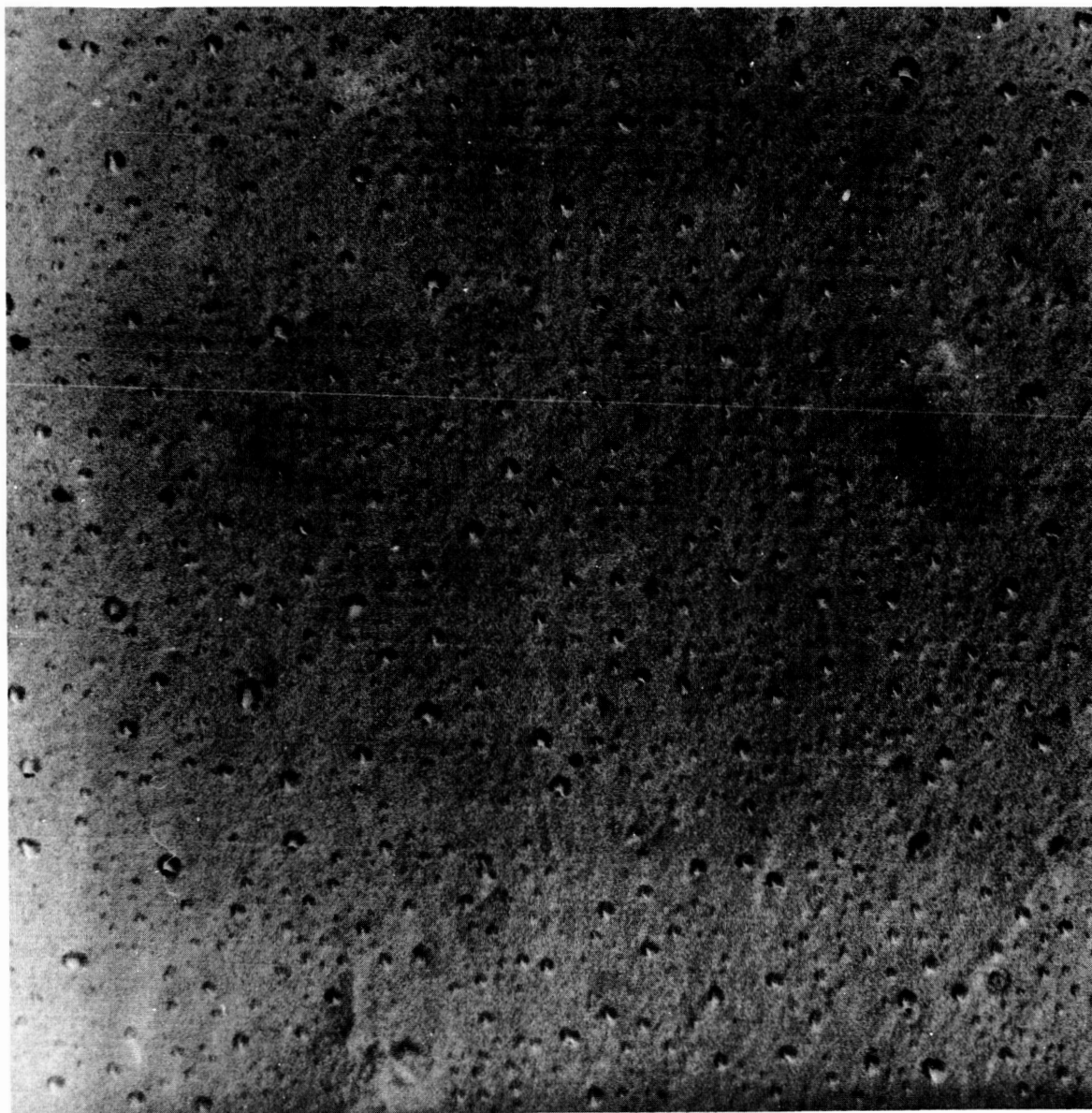
Fig. 27. Electron-micrograph of a gold surface transferred from optically-polished glass, for comparison with similar surfaces after gold and platinum flash coating.

Photographic Negative 220-4.



1μ

Fig. 28. Electron-micrograph of a gold surface transferred from optically-polished glass then flash coated with gold evaporated with an electron gun. Photographic Negative 220-10.



1 $\mu$

Fig. 29. Electron-micrograph of a gold surface transferred from optically-polished glass then flash coated with platinum evaporated with an electron gun.

Photographic Negative 220-20.



## PROGRESS ON TASK 2 OF CONTRACT EXTENSION

Task 2 of the extension of NONR 4277 (00)(X) was called, "Transfer Mirror Fidelity and Stability" in the proposed extension of NONR 4277 (00)(X) submitted by Bausch & Lomb with the date December 1964.

During the period July 1, 1965 to September 30, 1965, many sets of transfer films and Cr coatings were deposited. Each set of transfer films and of Cr coated glass blanks was reserved for the testing of one test cement and of a control cement (DER 322). The cements in Table III have been used in these tests. Transfer of the Cr-Au film from its original plate to the backing plate occurred without serious loss of smoothness visually as noted in Table III. Reflectances of one control transfer film and of the good mirrors were measured in September 1965. Electron-micrographs of one edge area will be made. The transfer films will be placed in our  $10^{-8}$  torr vacuum chamber for a minimum of two weeks, then reexamined for XUV reflectance and electron-micrograph appearance.

The final report of this work is scheduled for December 25, 1965. Task 3 of this project is called, "Grating Efficiency Correlation Continuation." It will use transfer films made during the Task 2 work. Test gratings ruled in these films will be evaluated. The starting date for this task is October 1, 1965. The report on it is scheduled for March 25, 1965. It will be the final work done on this contract.



Table III. Cements being tested for Task 2 program.

| <u>Cement</u>               | <u>Type</u>                    | <u>Transfer</u>        | <u>Appearance</u>               |
|-----------------------------|--------------------------------|------------------------|---------------------------------|
| DER 322<br>(fluid epoxy)    | Thermosetting<br>Hard epoxy    | Excellent<br>(Control) | Excellent                       |
| KD-194<br>(Al-filled)       | Thermosetting<br>Hard epoxy    | Excellent<br>scratches | Orange peeled                   |
| Bakelite                    | Thermosetting<br>Hard phenolic | Partial                | Blistered; not<br>cured         |
| HE-63<br>(Hawkeye)          | Thermosetting<br>Hard allyl    | Good                   | Slightly<br>clouded             |
| Balsam                      | Thermoplastic                  | None                   | Fractured<br>cement             |
| Cellulose<br>caprate        | Thermoplastic                  | Good                   | Fine blisters                   |
| Versalon<br>XR-1100         | Thermoplastic<br>polyamide     | Good                   | Air bubbles<br>(viscous cement) |
| Marco                       | Polyester                      | None                   |                                 |
| Lens Bond<br>(Frankfort)    | Polyester                      | Fair                   | Small blisters                  |
| Epocryl                     | Epoxy-Acrylic                  | None                   |                                 |
| Epocryl 50<br>(50% Styrene) | Epoxy-Acrylic                  | Poor                   | Clouded                         |
| Polyurethane<br>(fluid)     | Elastomer                      | Good                   | Fine orange peel                |
| Silicone<br>(primed)        | Elastomer                      | Good                   | Shear lines                     |
| Glass Resin                 | Inorganic                      | Fair                   | Crazed                          |
| Water Glass                 | Inorganic                      | Poor                   | Very poor                       |

## REFERENCES

..... RC468 5M-V865 Philips Electronic Instruments

..... Tech Bits 65-2, P-3 Eastman Kodak Company

Clark, G.L., The Encyclopedia of Microscopy, Reinhold Publ. 1961,  
Pp. 231-232.

Anderson, W., Griffin, G., Mooney, F., Wiley, R., Appl. Opt. 4,  
P. 999, August 1965.

Price, C.W., RFP-389, U.S. Atomic Energy Commission  
Contract AT (29-1) - 1106. October 1964.

Hunter, W. R., Proceedings of the Xth Colloquium Spectroscopicum  
Internationale, P. 247, 1962.

Edited by Lippincott and Margoshes.

Published by Spartan Books.

Canfield, L. R., Hass, G., and Hunter, W. R.,  
Jour. de Phys. 25 P. 124, 1964.